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THERMODYNAMIC PROPERTIES OF MOLYBDENUM AND COLUMBIUM: AN ANNOTATED BIBLIOGRAPHY

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Compiled by

JACK B. GOLDMANN

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Lockheed

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A GROUP DIVISION OF LOCKHEED AIRCRAFT CORPORATION

SUNNYVALE, CALIFORNIA

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ABSTRACT

The data which are included encompass heat of formation, activity, entrophy, diffusion rates and diffusion constants. In addition, references to the research which has been done and the tests and instrumentation is noted. The period covered by this bibliography is 1924–1962, with special emphasis on the literature published since 1955.

The resources of Lockheed Missiles & Space Company Technical Information Center were utilized in the preparation of this bibliography.

Search completed Dec 1962.

CONTENTS

1. Albrecht, W. M., Mallett, M. W. and Goode, W. D.
Equilibria in the niobium-hydrogen system.
ELECTROCHEMICAL SOCIETY. JOURNAL
105:219–223, Apr 1958.

Determined at 100–900° C., 10–1000 mm. of mercury hydrogen pressure, and atomic ratios of hydrogen to columbium of 0.01–0.85. Single-phase solid solution produced.

2. Albrecht, W. M. and Goode, W., Jr.
REACTION OF NITROGEN WITH NIOBIUM.
Battelle Memorial Institute, Columbus, Ohio.
BMI-1360. 6 Jul 1959. 13p.

Reaction rate of Cb with nitrogen is determined gravimetrically and volumetrically from 675 to 1600° C. Diffusion coefficients and terminal solubility for nitrogen in Cb are determined from 800 to 1600° C.

3. Allen, R. D., Glasier, L. F. and Jordan, P. L.
Spectral emissivity, total emissivity, and thermal conductivity of molybdenum, tantalum, and tungsten above 2300° K. JOURNAL OF APPLIED PHYSICS 31(8):1382–1387, Aug 1960

Arc-melted molybdenum was measured for emissivities and thermal conductivity from 2300° to melting. The spectral emissivity of molybdenum is constant and the total emissivity increased with temperature, while the thermal conductivity decreases. Methods are described and data are tabulated.

4. Alyamovskii, S. I., Gel'd, P. V. and Matveenko, I. I.
Concentration regions of niobium silicide stability at 1250° C. ZHURNAL NEORGANICHESKOI KHIMII 7:836–843, Apr 1962
(In Russian)

Phase studies of Nb-Si at 1500° C indicated two stable silicides alpha-Nb_5Si_3 and $NbSi_2$; Nb_4Si was not observed. It was found that alpha-Nb_4Si_3 is a special phase

with variable composition and stability ($NbSi_{0.58}$ to $NbSi_{0.66}$) at 1250°. The niobium bisilicide is a single phase ($NbSi_{1.85}$ to $NbSi_{2.20}$) developed from alpha-Nb_5Si_3 by replacement.

5. Amosov, V. M. and Dianov, V. V.
Investigation of the processes of pressing and high temperature sintering of electrolytic powders of tantalum and niobium. POROSHKOVAYA METALLY 3:14–19, May–Jun 1961 (In Russian)

A number of physicochemical properties of electrolytic powders of tantalum and niobium with a raised content of impurities were investigated in the process of pressing and high-temperature sintering in vacuum. The question of removing the impurities in the process of vacuum refining of tantalum and niobium is discussed. The results were used for developing production methods of obtaining plastic tantalum and niobium from electrolytic powders.

6. Argent, B. B. and Phelps, R.
The oxidation of niobium. INSTITUTE OF METALS. JOURNAL 88:301–304, Mar 1960.

Oxidation rate of pure Cb measured in dry and moist oxygen and air from 400 to 1050° C. Anomalies in oxidation rates are explained by departures from stoichiometry in the oxides.

7. Argent, B. B. and Milne, G. J. C.
The physical properties of niobium, tantalum, molybdenum and tungsten. JOURNAL OF THE LESS-COMMON METALS 2(2–4):154–162 Apr–Aug 1960.

A review of the physical properties of molybdenum as compared with other transition elements covers electronic structures, molar specific heat, vapor pressures and thermodynamic properties, among others.

8. Aylmore, D. W., Gregg, S. J. and Jepson, W. B.
Oxidation of niobium in the temperature range 350–750° C. ELECTROCHEMICAL SOCIETY. JOURNAL 107:495–501, Jun 1960.

Kinetics of Cb oxidation in dry and moist oxygen in the range 350–750° C. at 1 atm. and 0.1 atm. pressure were measured. At 350° the oxidation is protective, but at 400° and above the metal oxidizes. There is a rate transition at 400, 450 and 500°, but not at higher temperatures. Moisture decreases the rate at 400 and 450° but does not affect the rate at 600°. Purity of the sample and pressure also affect the rate.

9. Barrett, C. A. and Clauss, F. J.
Oxidation of columbium-chromium alloys at elevated temperatures. In TECHNOLOGY OF COLUMBIUM (NIOBIUM). N. Y., Wiley, 1958. p. 98–105.

Oxidation rates and scale characteristics were determined for Cb alloys containing 0.8 to 11.6% Cr at 800, 1000 and 1200° C. At 800° C., optimum Cr concentration for soundest scale and lowest oxidation rate was found at 5 to 8%. At 1000° C., scale characteristics and oxidation resistance improved with increasing Cr. At 1200° C., Cr had no appreciable effect in range studied.

10. Bartlett, E. S. and Williams, D. N.
Oxidation rate of molybdenum in air. AMERICAN INSTITUTE OF METALLURGICAL ENGINEERS. TRANSACTIONS 212:280, Apr 1958

Values at temperatures from 1400 to 2150° F.

11. Bartlett, E. S. and Houck, J. A.
PHYSICAL AND MECHANICAL PROPERTIES OF COLUMBIUM AND COLUMBIUM-BASE ALLOYS. Battelle Memorial Institute, Columbus, Ohio. DMIC Report 125
22 Feb 1960. 68p.

Data on seven currently commercial or pilot-production alloys. Of these, the Cb-15%W-5%Mo-1%Zr-0.05%C-0.05%O composition appears to be the most promising for elevated-temperature structural applications, based on strength and recrystallization temperatures. This alloy has a 100-hr. rupture stress of 35,000 psi. at 2000° F., or 17,000 psi. at 2200° F.

12. Begley, R. T., ed.
DEVELOPMENT OF NIOBIUM-BASE ALLOYS.
Westinghouse Electric Corp., Aviation Gas Turbine Div., Kansas City, Mo. Period covered 1 Jun 1957 to 1 Jun 1958. 30 Nov 1958. 192p.
[Contract AF33(616)-3316] [WADC TR-57-344 (Pt. II)] ASTIA AD-155 583.

Flow and fracture studies indicate that the ductile-to-brittle transition of niobium is little affected by oxygen content in the range 0.01 to 0.1% O_2. The impact transition range of electron-beam-melted niobium was found to be considerably below that of powder metallurgy niobium of somewhat higher oxygen concentration. Strain hardening and strain aging in niobium were studied. An activation energy of 27,100 cal/mole was determined for strain aging in niobium. The recrystallization behavior of electron-beam-melted niobium was studied in detail. Creep-rupture data were obtained on electron-beam-melted niobium at 871 and 982° C. The electron-beam material had much lower rupture strength than powder metallurgy niobium containing 0.6%Zr tested previously. The effect of temperature on the modulus of elasticity of niobium and tantalum was determined in the range 25 to 900° C. Studies of the thermodynamics of niobium oxides and the kinetics of Nb-water vapor reaction were carried out. The thermodynamic functions obtained in this study for the formation of Nb_2O_5 from Nb_2O_4 are in excellent agreement with calculated values. A selection of the most promising weldable niobium-base alloys was made on the basis of available physical and mechanical property data. Niobium of low oxygen and nitrogen content was produced by cage-zone and floating-zone melting techniques. Evaluation of material prepared by electron-beam melting and vacuum arc melting was carried out.

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13. Begley, R. T., ed.
DEVELOPMENT OF NIOBIUM-BASE ALLOYS.
Westinghouse Electric Corp., Aviation Gas Turbine Div., Kansas City, Mo. Period covered 1 Jan 1956 to 1 Mar 1957. Nov 1957. 112p. [Contract AF33(616)-3316] [WADC-TR-57-344] ASTIA AD-155 583.

The flow and fracture characteristics of commercial purity powder metallurgy niobium were investigated in the range 250 to -196° C. Niobium was found to undergo a ductile-brittle transition in the range -125 to -196° C, and the transition temperature range of niobium was found to be less affected by the presence of interstitial impurities than many other body-centered cubic metals. The creep-rupture properties of powder metallurgy niobium were investigated at 982 and 1093° C (1800 and 2000° F), and the 100-hour rupture strength of commercial niobium in vacuum were determined to be significantly greater than unalloyed molybdenum. The creep-rupture results suggest that small quantities of gaseous contaminants may be responsible for the high strength of commercial niobium at elevated temperatures. The oxidation behavior of niobium was investigated in the temperature range 350 to 700° C. At the higher temperatures, oxidation followed a linear rate law. Between 500 and 625° C, the rate of oxidation was found to be nearly independent of temperature. Oxygen and nitrogen contamination of welding atmospheres was studied to determine its effect on the weld properties of niobium. Nitrogen was established to be very detrimental to the mechanical properties of niobium welds. High-purity niobium, having a hardness of less than 60 VPN, was produced by cage-zone refining techniques.

14. Berkowitz, J.
KINETICS OF OXIDATION IN THE Mo-Si SYSTEM. Arthur D. Little, Inc., Cambridge, Mass. Final rept. Sep 60—Sep 61 on Refractory, Inorganic and Non-Metallic Materials. May 1962. 42p. [Contract AF33(616)-6154] (ASD TDR 62-203, pt. 1) ASTIA AD-278 826.

The oxidation behavior of Mo, Mo_3Si, Mo_5Si_3, and $MoSi_2$ was studied at temperatures between 1000 and 1725° C at oxygen partial pressures less than 20 mm. Pure Mo metal oxidizes linearly in this range, with a ratio constant k = k(sub o)1(-22,000/RT)p(0.87), where p is oxygen partial pressure, the gas constant R is in cal/mole, T is in K, and k (sub o) is a constant independent of temperature and pressure. To

a good approximation, oxidation of the Mo silicides follows a logarithmic rate law, Q = Q (sub o) (1-1) (-alpha t), where Q is the total oxygen consumed between time zero and time t, while Q (sub o) and alpha are constants, dependent upon composition, pressure and temperature. At any fixed temperature, the most oxidation resistant material is $MoSi_2$; Mo_5Si_3 is oxidized to a larger extent before a protective coating is established, and Mo_3Si is oxidized still more extensively. The rates of oxidation of all 3 compounds eventually reaches a low-limiting value. Metallographic examinations established that oxidation proceeds at least in part by diffusion of oxygen inward. An apparatus utilizing a thermal conductivity bridge is described for continuous measurement of the rate of oxidation of metallic materials at temperatures between 900 and 2100° C.

15. Berkowitz, J., Inghram, M. G. and Chupka, W.
Mass spectrometric study of molybdenum oxide vapor. AMERICAN PHYSICAL SOCIETY. BULLETIN Ser.2.1(4):202, 26 Apr 1956 (Abstract)

The molecular species evaporting from a crucible of MoO_3 were studied spectrometrically. In the range 700° – 950° K the main species are Mo_3O_9, Mo_4O_{12} and Mo_5-O_{15}; the enthalpy values are about 80, 93 and 110 kcal/mole, respectively. Earlier work is contradicted.

16. Berkowitz, J.
STABILITY OF CERAMIC MATERIALS AT TEMPERATURES TO 2000° C. Little, Arthur D., Inc., Cambridge, Mass. Report Jan 59 – Jan 60 on Ceramic and Cermet Materials Development. Sep 1960. 59p. [Contract AF 33(616)-6154, Proj. 7350] [WADD TR 60-377] ASTIA AD-248 965.

A study was made of the oxidation resistance of a number of commercial samples of $MoSi_2$ and WSi_2 in the temperature range 1600 – 1700° C. A thermal conductivity technique was developed to measure oxygen pick-up by the samples continuously. From the thermal conductivity measurement and the net weight change of the samples, an estimate was made of the rates of formation of volatile and non-volatile oxides. On the basis of x-ray analysis, oxidation products are believed to be volatile XO_3 (vapor) and non-volatile glass of the form SiO_2. nXOm, where X = Mo or W; n and m are not known. Tests were conducted at a total pressure of one atmosphere in a helium stream containing about 10 mm of oxygen and flowing at 119 cc/min. The oxidation of WSi_2 was also studied in a helium stream containing 5 mm of water vapor. The principal solid product in this case was metallic tungsten.

17. Bolef, D. I. and de Klerk, J.
Elastic constants of single-crystal Mo and W between 77° and 500°K. JOURNAL OF APPLIED PHYSICS 33(7):2311–2314, Jul 1962.

The temperature-dependence of the elastic constants of molybdenum monocrystals from 77°-500°K and data are presented in a table. Shear anisotropy is anomalously low and does not depend on temperature.

18. Bolgar, A. S., Verkhoglyadova, T. S., and Samsonov, G. V.
Vapor pressure and rate of evaporation of some refractory compounds in vacuum at high temperatures. AKADEMIIA NAUK SSSR. IZVESTIIA. OTDELENIE TEKHNICHESKIKH NAUK. METALLURGIIA I TOPLIVO. 1:142–145, Jan–Feb 1961 (In Russian)

Evaporation and vapor pressures were determined by the Langmuir method over the temperature range 1100° –2000° C for various refractory compounds, including TiC, TiB_2, TiN, ZrC, ZrB_2, HfC and $MoSi_2$. Heats of evaporation of these compounds were also calculated. In the temperature range investigated, all compounds studied evaporated as molecular complexes. Vapor pressure and evaporation rate data are tabulated.

19. Booker, J., Paine, R. M. and Stonehouse, A. J.
INVESTIGATION OF INTERMETALLIC COMPOUNDS FOR VERY HIGH TEMPERATURE APPLICATIONS. Brush Beryllium Co., Cleveland, Ohio. Dec 1960. 143p. [Contract AF 33(616)-6540] [WADD-TR-60-889] ASTIA AD-265 625.

...disilicides of Mo were screened for compounds capable of serving as structural materials at temperatures above 2500° F. The compounds studied were ... $MoSi_2$...

20. Brauer, G. and Jander, J.
The nitrides of niobium. ZEITSCHRIFT
FUER ANORGANISCHE UND ALLGEMEINE
CHEMIE 270:160–178, 1952 (In German)

Pure metallic niobium and pure nitrogen produced NbN when heated together for four to five hours at atmospheric pressure to 1300–1450°. NbN containing up to 25% oxygen was formed when small amounts of oxygen entered the reaction mixture. The two compounds differ only insignificantly in crystal structure. The density of pure niobium nitride is 8.58.

21. Braun, H. and Sedlatschek, K.
Recent investigations of high temperature alloys
of molybdenum and tungsten. JOURNAL OF
METALS 12(5):377–378, May 1960.

Molybdenum alloys with rhenium and transition metals are being studied by many agencies. Additions of niobium or tantalum up to 100% have been surveyed technologically, and their density, hardness, electrical resistivity, thermoelectric force, corrosion resistance and workability have been evaluated. The V-W and Hf-W systems were examined by metallography and x-ray diffraction, and their phase diagrams were established. This paper was presented before the International Powder Metallurgy Conference in New York, 13–17 June 1960.

22. Brewer, L., et al.
High melting silicides. AMERICAN CERAMIC
SOCIETY. JOURNAL 33:291–294, 1950.

The tantalum-silicon, molybdenum-silicon, and tungsten-silicon systems have been investigated for solid phases stable above 1900° K. Three new tantalum silicides phases, two new molybdenum silicide phases, and one new tungsten silicide phase are reported besides the MSi_2 phases previously known. The crystal structure of Mo_3Si is described. Lower limits for eutectic temperatures are given for these three systems, and the relative stabilities of metallic silicides of groups III through VII are discussed.

23. Brewer, L. and Krikorian, O.
Reactions of refractory silicides with carbon and nitrogen. ELECTROCHEMICAL SOCIETY. JOURNAL 103(1):38–51, 1956.

Silicides of ... and Nb were investigated to determine the phases present around 2000° K. ... Reactions of silicides of Nb, ... Mo... with carbon were studied at these temperatures, and a limited amount of work was done on the reactions of some silicides with nitrogen. The data were used to establish ternary phase diagrams for the systems and to obtain upper and lower limits for heats of formation of the silicides.

24. Bridges, D. W. and Fassell, W. M., Jr.
High pressure oxidation of niobium. ELECTROCHEMICAL SOCIETY. JOURNAL 103:326–330, Jun 1956.

Niobium oxidizes according to the linear rate law from 400–800° C. The oxidation rate is extremely pressure sensitive above 550° C.

25. Bryant, C. A. and Keeson, P. H.
Low temperature specific heat of molybdenum. JOURNAL OF CHEMICAL PHYSICS 35(3):1149–1150, Sep 1961

Molybdenum powder was arc melted into a pellet and sintered into a rod, and the specific heat was measured in the 1°–4.5° K range. The difference in results is discussed, and the best value is accepted as that from the denser and more pure rod specimen. Data are tabulated.

26. Bucher, E., Heiniger, F. and Mueller, J.
Superconductivity and paramagnetism of complex phases of transition metals. HELVETICA PHYSICA ACTA 34(8):843–858, 30 Dec 1961 (In German)

The alloys Ti-Re, Mo Re, Mo-Ru, Mo-Os and Mo-Ir, of the composition A_xB_{1-x} (A = Ti, Mo) and having the structure of σ phase and a-Mn phase, were found to

be superconducting above 1.2°K. Their paramagnetic susceptibility is independent of the critical temperature of superconductivity. Its order of magnitude is comparable with that of the susceptibility of molybdenum and rhenium.

27. Calverley, A., Mendelssohn, K. and Rowell, P. M.
Some thermal and magnetic properties of tantalum, niobium, and vanadium at helium temperatures.
CRYOGENICS 2(1):26–33, Sep 1961

Thermal conductivities of vanadium monocrystals were compared with that of severely work-hardened polycrystalline metal at liquid helium temperatures. The last approached theoretical values more closely than the monocrystal curves, which behaved imperfectly because of impurity and crystal imperfections. Graphs are shown.

28. Campbell, J. E. et al
INTRODUCTION TO METALS FOR ELEVATED-TEMPERATURE USE. Battelle Memorial Institute. Defense Metals Information Center, Columbus, Ohio. Report DMIC-160. 27 Oct 1961
92p. [Contract AF 18(600)-1375]

The general categories of metals suitable for application at temperatures in excess of 800°F are discussed, along with the general advantages and limitations of each. Some generalized mechanical and physical property data are included, and the problems of reactions at elevated temperatures are briefly discussed.

29. Carter, W. J.
THERMODYNAMIC PROPERTIES OF SEVEN METALS AT ZERO PRESSURE. Los Alamos Scientific Laboratory, New Mexico. Report LAMS-2640. Sep 1961. 62p. [Contract 7405-Eng-36]

Figures are presented which summarize the data presently available in the literature on the variation of thermal expansion and specific heat with temperature for the seven metals Cu, In, Nb, Pd, Pt, Ta, and Zt in the temperature range 293 to 3000° K. The best guesses for the function a (T) and $C_p(T)$ were used to prepare curves and polynomial fits of heat content and temperature as functions of volume which are needed in shock wave equation of state work.

30. Cathcart, J. V. and Young, F. W.
Influence of reactor radiation on the oxidation of niobium. CORROSION 17:77–79 Feb 1961.

No measurable change is detected in the rate of oxidation or in the nucleation process associated with the oxidation of Cb. On the basis of estimates of the extent of radiation damage in thin oxide films, it is concluded that the damage resulting from irradiation at currently available neutron flux levels is unlikely to change the number of oxide lattice defects sufficiently to produce a significant change in the oxidation rate.

31. Chambers, R. H. and Schultz, J.
Dislocation relaxation spectra in plastically deformed refractory B. C. C. metals. ACTA METALLURGICA 10(4):466–483, Apr 1962.

An internal friction study revealed two broad thermally activated relaxation peaks in molybdenum plastically deformed at 25° C. A post-relaxation rise is observed when a small amount of interstitials is present. Only a small fraction of the dislocations in cold-worked molybdenum is free to move at 5° K, indicating the necessity of thermal activation over barriers larger than those in f. c. c. metals. Graphs are shown.

32. Clark, C. B.
Calculated lattice specific heats for seven BCC elements. PHYSICAL REVIEW 125(6):1898–1902, 15 Mar 1962.

Specific heats were calculated for molybdenum and vanadium from reported elastic constants. Corresponding Debye characteristic temperatures are plotted.

33. Clauss, F. J. and Barrett, C. A.
Preliminary study of the effect of binary alloy additions on the oxidation resistance of columbium. In TECHNOLOGY OF COLUMBIUM (NIOBIUM). N.Y., Wiley, 1958. p. 92–97.

Addition elements, Ti, V, Cr, Zr, Mo, Ta, W, Fe, Co, Ni, Ir, Al, Si, Cu, Ge, Se and Re were added singly in 1, 2, 3, 5, 10 and 25 at . % and specimens were fabricated by powder metallurgy. Nature of scale and resistance to oxidation were evaluated by continuous exposure to dried air for 4 hr. at 1000° C. and 2 hr. at 1200° C.

34. **Danneberg, W. and Krautz, E.**
Study of autodiffusion in molybdenum.
ZEITSCHRIFT FUER NATURWISSENSCHAFTEN
16a(9):854−857, Sep 1961 (In German)

The temperature dependence of autodiffusion in thin molybdenum wire was measured at 1600° −2180° C with the aid of the radioactive Mo^{99}. The results confirmed the hypothesis of lattice vacancy displacement as the mechanism of autodiffusion in molybdenum.

35. Davis, M.
Molybdenum and tungsten; possibilities in the nuclear field. NUCLEAR ENGINEERING
3(27):243−246, Jun 1958

High temperature and other properties of molybdenum and tungsten, which may make them attractive as canning materials for some types of advanced nuclear reactors; problem of development of suitable alloys.

36. Dillaway, R. B.
COMPATIBILITY OF MATERIALS WITH HIGH TEMPERATURE POTASSIUM. Rocketdyne, Canoga Park, Calif. Third Quarterly Progress Report, 1 Nov 1960 through 31 Jan 1961. Report R-2617-3. 3 Mar 1961. 62p. (NASA contract NAS5-453)

This report describes the third quarter of effort in a 13-month evaluation of refractory metals for potential use in high-temperature, turboelectric, space power plants using potassium as the working fluid. Construction of two columbium-1 percent zirconium loops is entering the final phases. Changes in boiler design will be made in furtherance of experience with operation of the heater units. Operation of the low temperature loop fabricated of super alloys has been terminated; the results have been compiled. Capsule studies have moved from bimetal systems to all-columbium alloy. Coupling of nickel alloys with columbium alloys in potassium resulted in metallic deposits on the nickel alloys and decarburization of the nickel alloys by columbium. Columbium alloy capsule experiments disclosed little corrosion up to 2200° F.

37. Douglas, T. B. and Logan, W. M.
Heat content of molybdenum disilicide from
0° to 900°C. JOURNAL OF RESEARCH OF
THE NATIONAL BUREAU OF STANDARDS
53:91–93, 1954 (NBS Research Paper No. 2520)

The heat content relative to 0°C of a sample of molybdenum disilicide ($MoSi_2$) was measured at 100-degree intervals up to 900°C, using a nichrome-V container for the sample, a silver-core furnace, and a precision ice calorimeter. Correction were applied for the impurities (approximately 2 percent) reported to be in the sample. The equation

$$H_t - H_o = 0.48560t + 3.586(10^{-5})t^2 - 41.78 \log_{10} [(t + 273.16)/273.16] ,$$

giving between 0° and 900°C the heat content in absolute joules per gram at t° less that at 0°C, represents the unsmoothed corrected mean values, with an average deviation of 0.24 absolute joule gram $^{-1}$. This equation is believed to have an absolute accuracy within 2 percent between 100° and 800°. Values of the relative heat content, heat capacity, and relative entropy derived from this equation are formulated and tabulated at even temperatures.

38. Douglas, T. B. and Logan, W. M.
THERMAL CONDUCTIVITY AND HEAT
CAPACITY OF MOLTEN MATERIALS. PART 3.
THE HEAT CAPACITY OF MOLYBDENUM
DISILICIDE FROM 0° to 900°C. National
Bureau of Standards, Washington, D. C.
WADC Technical Report No. 53-201, Pt. 3.
1953. 13p. [Contract AF33(616)52-10]
ASTIA AD-24 019.

A new apparatus is described for determining at high temperatures the thermal conductivities of liquids and solids having relatively high conductivities. The apparatus employs steady state longitudinal heat flow along a rod surrounded by a matched guard tube. Results of molybdenum disilicide are described.

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A new apparatus is described for determining at high temperatures the thermal conductivities of liquids and solids having relatively high conductivities. The apparatus employs steady state longitudinal heat flow along a rod surrounded by a matched guard tube. Results of molybdenum disilicide are described.

39. Douglass, R. W.
PHYSICAL AND MECHANICAL PROPERTIES OF MOLYBDENUM AND THE Mo-0.5Ti ALLOY. Battelle Memorial Institute, Columbus, Ohio. DMIC Memorandum 14. 10 Apr 1959. 21p.

The mechanical properties reported are representative of hot-cold worked and/or stress-relieved molybdenum or Mo-0.5Ti, except for those obtained above 3000°F. Data are tabulated for room- and high-temperature tensile properties, modulus of elasticity, ductility, and ductile-to-brittle transition. Stress-strain curves up to 2800°F at various holding times in air and argon are presented. Mo-0.5Ti has equal ductility and the better tensile strength.

40. Eldridge, E. A. and Deem, H. W.
Molybdenum and its alloys. In REPORT ON PHYSICAL PROPERTIES OF METALS AND ALLOYS FROM CRYOGENIC TO ELEVATED TEMPERATURES. American Society for Testing Materials. STP-296. 1961. p. 143-157

Thermal conductivity is plotted for molybdenum, Mo-0.5Ti, Mo-2.5Ti, Mo-5.0Ti, Mo-0.005Fe, Mo-0.3Nb, and Mo-0.016C. Electrical resistivity is plotted for Mo-0.016C and Mo-0.019C. Forty-six references to other properties of molybdenum and molybdenum alloys in various heat treated conditions are presented.

41. Evans, J. E., Jr.
THERMAL CONDUCTIVITY OF 14 METALS AND ALLOYS UP TO 1100°F. (595°C). National Advisory Committee for Aeronautics, Washington, D. C. NACA RM E50L07. 1951. 15p.

The thermal conductivity of 14 metals and alloys was determined in temperature ranges having a maximum of 1100°F. The metals included...molybdenum disilicide... The...high-temperature alloys...with one exception, have conductivities that increase with increasing temperature...molybdenum disilicide...after heat treatment, have condutivities that decrease as the temperature is increased... A comparison method was used to obtain the results in which the thermal conductivity of the test sample was compared with the conductivity of high-purity lead.

39. Douglass, R. W.
PHYSICAL AND MECHANICAL PROPERTIES OF MOLYBDENUM AND THE Mo-0.5Ti ALLOY. Battelle Memorial Institute, Columbus, Ohio. DMIC Memorandum 14. 10 Apr 1959. 21p.

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42. **Ewing, C. T. and Baker, B. E.**
THERMAL AND RELATED PHYSICAL PROPERTIES OF MOLTEN MATERIALS. PART I. THERMAL CONDUCTIVITY AND HEAT CAPACITY OF MOLYBDENUM DISILICIDE. **U.S. Naval Research Laboratory, Washington, D. C. WADC Technical Report No. 54-185, Pt. 1. 1954. 27p. ASTIA AD-50 565.**

Thermal conductivity and heat capacity figures for hot-pressed molybdenum disilicide are reported to 840°C. The apparatus and method employed for each property study are described in detail. The conductivity coefficients were measured in a longitudinal type system with guard-ring compensation; the heat capacity results were derived from enthalpy measurements made by a drop-method with a copper block calorimeter. A detailed analytical description for each molybdenum disilicide test sample is included, and the change in property value from ideal due to the impurity content of physical form of the test sample is predicted, where possible.

43. Fawcett, E.
Magnetoresistance of molybdenum and tungsten. AMERICAN PHYSICAL SOCIETY. BULLETIN 7(3):231, 26 Mar 1962. (abstract)

An anisotropy in the transverse magnetoresistance of molybdenum is observed at 4.2°K in a field of 18 kgauss. When the current direction is near <100>, <110> and <211>, a magnetoresistance ratio of several hundred is shown with a smooth variation by a factor about two as the field is rotated. The interpretation that there are no cyclotron orbits establishes the itinerant character of s and d wave functions.

44. Fieldhouse, I. B., Hedge, J. C. and Lang, J. I.
MEASUREMENTS OF THERMAL PROPERTIES, 1 JULY 1957 TO 31 MARCH 1958. Illinois Institute of Technology, Chicago. Armour Research Foundation. Report WADC-TR-58-274. 1 May 1958. 87p. [Contract AF33(616)-3701] ASTIA AD-206 892.

The materials investigated were...niobium...The thermal conductivity, specific heat, and linear thermal expansion were measured from 100 to 3000°F, or to the

42. **Ewing, C. T. and Baker, B. E.**
THERMAL AND RELATED PHYSICAL PROPERTIES OF MOLTEN MATERIALS. PART I. THERMAL CONDUCTIVITY AND HEAT CAPACITY OF MOLYBDENUM DISILICIDE. U.S. Naval Research Laboratory, Washington, D.C. WADC Technical Report No. 54-185, Pt. 1. 1954. 27p. ASTIA AD-50 565.

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The materials investigated were...niobium...The thermal conductivity, specific heat, and linear thermal expansion were measured from 100 to 3000°F, or to the

melting point of the material, whichever was lower. Both the experimental measurements and the results of the conversion of these measurements to the desired physical properties are given.

45. Freeman, R. R.
Properties and applications of commercial molybdenum and molybdenum alloys. In THE METAL MOLYBDENUM. Harwood, J. J., ed. Cleveland Ohio, American Society for Metals, 1958. p. 10–30.

Compared with other metals, molybdenum has generally higher modulus of elasticity, thermal and electrical conductivities, and high-temperature creep rupture strength. Coefficient of expansion and neutron absorption cross section are low, while mechanical properties depend on the work done below the recrystallization point. Corrosion resistance to liquid alkali and other metals is good, but to oxidizing agents is poor. Commercial availability of forms of the arc-cast and powder metallurgical metal are outlined. Applications to industry are described.

46. Friedman, R.
High-temperature durability of molybdenum in oxygen-deficient combustion gases. JET PROPULSION 24:187, May–June 1954.

Test data for uncoated molybdenum at temperatures up to 2600°F. Corrosion rates can be tolerated in many applications.

47. Geach, G. A. and Jones, F. O.
Interactions in mixtures of hard materials at very high temperatures. In PLANSEE PROCEEDINGS 1955. Benesovsky, F., ed. London, Pergamon, 1956. p. 80–91.

Previous work in the Associated Electrical Industries Research Laboratory has shown melting in an arc-furnace of the cold-hearth non-consumable electrode type to be a technique of value in the study of hard-materials. It enables melting points of mixtures to be measured quickly and materials can be prepared free from contamination to which some hard materials are sensitive. Several binary mixtures of carvides, boride and silicides have been prepared and examined. Appreciable interaction has so far been found only when one of the compounds used contained molybdenum.

48. Geballe, T. H. et al.
Superconductivity in molybdenum. PHYSICAL REVIEW LETTERS 8(8):313, 15 Apr 1962.

Pure molybdenum becomes superconducting at about 1°K, either with arc-melted or electron-beam-melted, floating-zone-refined crystals. Present results and those reported by others are tabulated. 2 references.

49. Gemmell, G. D.
Some effects of alloying on the strength properties of columbium at elevated temperatures. AIME. METALLURGICAL SOCIETY. TRANSACTIONS, 215:898–901, Dec 1959.

Effects of solid-solution alloying with titanium, molybdenum, and tungsten up to 10 wt.% solute on the strength of pure niobium at elevated temperatures (mainly 2000°F) were investigated by means of creep-rupture, hot-tensile, and hot-hardness testing and recrystallization studies.

50. General Electric Co. Flight Propulsion Laboratory Department, Cincinnati, Ohio.
REFRACTORY ALLOYS FOR USE AS STRUCTURAL MATERIALS IN RE-ENTRY OR BOOST GLIDE VEHICLES. Quarterly report no. 1, 1 Jun–1 Sep 1959. 126p. [Contract AF33(616)-6578]

The major portion of an alloy survey, made to determine candidate alloys for the construction of a structural component of a re-entry vehicle to be tested at 2500°F, was completed. Mechanical properties, recrystallization characteristics, and oxidation behavior are reported. One Nb alloy and three Mo alloys were identified that are semi-commercially available and that show potential for the above application. The Nb alloy F48 and the Mo alloy with the highest temperature capability, TZC, both require further processing development. Because of the desirable advantage the high strength Nb alloy has over Mo in oxidation resistance and reliability, and since two suitable Mo alloys (0.5Zr, and 0.5Ti-0.07Zr) are reasonably readily available, emphasis was placed on initiating process studies of F48 alloy: i.e., arc melting and sheet rolling. Considerable process information and development, including sheet bar extrusion, in-process coatings, and chemical analysis for Nb, acquired previously are reported. A survey for joining and fabrication procedures

for Nb and Mo alloys reveals the state of the art, with respect to Mo, is far advanced over Nb alloys. Strong ductile spot weld joints were obtained in one Nb alloy (82) compared to the brittle welds encountered with Mo. Welding, mechanical fasteners, brazing, forming, and machining are discussed. Although a comprehensive review of protective coatings uncovered a need for a large integrated program to solve the needs for "flight" coatings, there is no question that coatings are available to meet the requirements of this program.

51. Glasier, L. F., Jr., Allen, R. D. and Saldinger, I. L.
MECHANICAL AND PHYSICAL PROPERTIES OF
THE REFRACTORY METALS, TUNGSTEN,
TANTALUM, AND MOLYBDENUM ABOVE 4000°F.
Aerojet-General Corp., Azusa, Calif. Report
M-1826. Apr 1959. 89p.

Methods were developed and tests conducted on pure W, Ta, and Mo to provide mechanical and physical property data in the temperature range 2200 (4000°F) to 3300°C (6000°F). Data include modulus of elasticity, proportional limit, ultimate tensile strength, yield strength, prolongation, thermal expansion, thermal conductivity, and both total and spectral (0.65 μ) emissivities. It was found that the mean coefficients of linear thermal expansion for Mo, Ta, and W increase with temperature at an increasing rate. The total emissivities increase with temperature, usually at a decreasing rate, between 2300°F and the respective melting points, while Mo is constant within this temperature span. The thermal conductivities decrease linearly with temperature from 2300°F to the respective melting points.

52. Gleiser, M. et al
The properties of oxidation-resistant scales
formed on molybdenum-base alloys at elevated
temperatures. In SYMPOSIUM ON BASIC
EFFECTS OF ENVIRONMENT ON THE STRENGTH,
SCALING AND EMBRITTLEMENT OF METALS AT
HIGH TEMPERATURES. American Society for
Testing Materials. Philadelphia, The Society, 1955.
p. 65–86. (ASTM Special Technical Publication No. 171)

Various molybdates and molybdenum-containing oxide complexes were tested for stability at elevated temperatures to determine if they would be stable at operating temperatures and possibly present a barrier to further oxidation.

53. Glenny, E. and Taylor, T. A.
High-temperature properties of ceramics and cermets. POWDER METALLURGY 1-2:189-226, 1958.

The major properties of ... $MoSi_2$... are evaluated (creep strength, fatigue strength, thermal fatigue, oxidation resistance, and impact resistance). The equipment for studying alternating and steady mechanical stresses up to 1200°C. is described.

54. Goldschmidt, H. J.
A high-temperature x-ray investigation of niobium pentoxide and some problems concerning the oxidation of niobium. INSTITUTE OF METALS. JOURNAL 87:235-239, Mar 1959.

Phase transformations of columbium (niobium) pentoxide. The total oxidation of initially pure Cb metal is followed in sequence, the isothermal stages extending from solid solution of oxygen in Cb to Cb_20_5. Preferred orientation can be produced in thin films of alpha Cb_20_5, as distinct from the random loose oxide. The alpha or beta form of Cb_20_5 can be stabilized by additions of certain oxides of other metals.

55. Gonser, B. W. and Sherwood, E. M., eds.
TECHNOLOGY OF COLUMBIUM (NIOBIUM); PAPERS PRESENTED AT THE SYMPOSIUM ON COLUMBIUM (NIOBIUM) OF THE ELECTRO-CHEMICS AND METALLURGY DIVISION OF THE ELECTROCHEMICAL SOCIETY, MAY 15 AND 16, 1958, WASHINGTON, D.C. N.Y., Wiley, 1958. 127p.

Niobium is a high melting metal whose compounds are in general nonbasic in character. It forms refractory binary compounds with elements of Groups III, IV, V. Its melting point and reactivity impose restrictions on methods of preparation. The metal is of particular interest as a high temperature material of construction because of its strength at high temperatures and its workability. The sources, economic aspects, and supply situation are described. A review of the extractive metallurgy is presented. A solvent extraction system is presented for separating Ta and Nb. The preparation

of high-purity Nb by electrolytic processes is described. Analytical methods are presented for determining impurities in Nb. Tensile properties and rolling textures are presented for Nb sheet. The electroplating of metals on Nb is discussed. The development of oxidation-resistant Nb alloy is discussed.

56. Goodwin, H. B.
The world's hottest alloys. Parts I and II.
INDUSTRIAL RESEARCH MAGAZINE
1(3 and 4), 1959, 11p.

Some properties of the stable elements which have melting points above 3000°F are presented. These include molybdenum, tantalum, niobium, rhenium, chromium, the platinum group, and metal-ceramic composites. These "scarce" metals are found to exist in the earth's crust sometimes more than the more "common" metals, such as copper, lead, etc. Where copper and lead are found to exist in deposits, the "scarce" metals are found to exist as complexes and are scattered throughout the earth's crust. The most promising of these are niobium, molybdenum, tantalum and tungsten. A molybdenum wire when heated above 2000°F will oxidize visually, but when it is coated with disilicide it does not oxidize appreciably. A niobium-45% zirconium-5% titanium alloy was recently developed which is stated to be both oxidation resistant and fabricable. A combination of alloying and coating may be the best solution to the oxidation problem, with alloying serving to prevent coating defects from becoming catastrophic failures...

57. Greiner, E. S. and Buehler, E.
High-temperature plastic deformation of V_3Si and Nb_3Sn. AMERICAN PHYSICAL SOCIETY. BULLETIN 7(4):310, 23 Apr 1962. (abstract)

Stresses of 10,000 psi at 1650°C were used for 10–20% plastic deformation of V_3Si. The crystals were prepared by cooling from the peritectic melt (2050°C). The effect of deformation on critical current vs. magnetic field is reported. (Abstract)

58. Grimm, T. C.
EVALUATION OF PFAUDLER COATING FOR OXIDATION PROTECTION OF COLUMBIUM ALLOYS. McDonnell Aircraft Corp., St. Louis, Mo. Final report. Report no. 8886. 10 Jul 1962. 20p. [Contract AF 33(657)-7749] ASTIA AD-277 775.

Portions of the Asset vehicle which attain temperatures above 2500°F will necessitate the use of refractory metals and associated protective coatings. A Pfaudler disilicide coating appears promising for this purpose. Pfaudler reported that their two-step disilicide coating provides oxidation production for columbium alloys for periods of one hour at 3100°F. Eight Pfaudler coated columbium alloy specimens, seven Cb-1Zr and F-48, were tested for oxidation resistance in the temperature range of 2800-3200°F. Five of these specimens were tested using an oxy-acetylene torch with an excess of oxygen, and three were plasma jet tested. The Pfaudler disilicide coating protected the columbium alloys from gross oxidation in the temperature range of 2800-3200°F for the 15 minutes test periods employed.

59. Grinthal, R. D.
EVALUATION OF NEW HIGH TEMPERATURE MATERIALS. American Electro Metal Corp., Yonkers, N.Y. Progress report no. 2, Dec 1955 – Feb 1956. 23p. [Contract No. AF 33(616)-3198] ASTIA AD-89 650.

Results indicate that copper additions to molybdenum disilicide tend to decrease room temperature strength, increase electrical resistivity and lower oxidation resistance at elevated temperatures. Several compositions in the chromium-molybdenum-silicon ternary system have been arc melted and identified by x-ray-diffraction methods. Powder is presently being reacted which corresponds with the arc melted compositions, and which will be fabricated into test samples by powder metallurgical methods.

60. Grinthal, R. D.
NEW HIGH TEMPERATURE INTERMETALLIC MATERIALS. Firth Sterling Inc. American Electro Metal Div., Yonkers, N.Y. Period covered: 2 Sep 1955 through 2 Nov 1956. Nov 1956. 65p. [Contract AF33(616)-3198] (WADC-TR-53-190(Pt. 5) ASTIA AD-110 684.

Six ternary systems were investigated as to physical properties, oxidation resistance, mechanical properties, phase studies, and electrical resistivity. They were: Cu, Cr, and Ti with Mo-Si...

61. Gulbransen, E. A. and Andrew, K. F.
Kinetics of the reactions of columbium and tantalum with 0, N, and H. JOURNAL OF METALS 188:586–599, Mar 1950.

Reactions were studied in high vacua, using the vacuum microbalance method. Rates of several reactions are compared with other metals.

62. Gulbransen, E. A. and Andrew, K. F.
Oxidation of niobium between 375 and 700°C. ELECTROCHEMICAL SOCIETY. JOURNAL 105:4–9, Jan 1958.

Complex reaction rate varies with temperature; studied by the vacuum microbalance method.

63. Gulbransen, E. A. and Andrew, K. F.
Reactions of zirconium, titanium, columbium, and tantalum with the gases, oxygen, nitrogen, and hydrogen at elevated temperatures. ELECTROCHEMICAL SOCIETY. JOURNAL 9:364–376, Dec 1949.

A systematic study of the kinetics of the above reactions. Data are correlated with fundamental diffusion and solution processes.

64. Hägg, G. and Magneli, A.
Recent structure investigations of oxygen compounds of molybdenum and tungsten. REVIEWS OF PURE AND APPLIED CHEMISTRY 4(4):235–250, Dec 1954.

Mo_3O differs structurally from other molybdenum oxides since the low oxygen content does not permit oxygen polyhedra to coordinate around the metal atoms. The other oxides show an ability of the polyhedra to arrange in several ways; a similarity to the V-0 system is cited in which there are 12 phases. Pure molybdenum oxides do not have extended homogenity ranges. Atomic distances are about the same in all the oxide structures, except for the puckered metal atom layers in Mo_8O_{23} and Mo_nO_{3n-1} members. The tendency of five-coordination is greater in molybdenum than in tungsten.

65. Ham, J. L.
An introduction to arc-cast molybdenum and its alloys. AMERICAN SOCIETY OF MECHANICAL ENGINEERS. TRANSACTIONS 73:723–731, Aug 1951; Disc.: 731–732.

The general properties of unalloyed cast molybdenum, curves show the room-temperature tensile properties, hardness, and notched-bar values after annealing at various temperatures, the effect of testing temperature on hardness, and the transition temperatures for various types of impact tests. The need for addition of alloying elements. The effects of several of the alloying elements on the rate of work hardening and on the temperatures of softening and recrystallization were studied using a series of alloys which were extruded.

66. Harwood, J. J.
Protection of molybdenum against high-temperature oxidation. In THE METAL MOLYBDENUM. Cleveland, American Society for Metals, 1958. p. 420–461.

Most successful coating compositions that have been developed thus far center around the Ni-Cr alloy system. Both electroplated Cr-Ni layers and sprayed Ni-Cr alloys containing S and B have demonstrated a capacity to protect Mo up to about 2000°F. Sprayed mixed silicide layers also appear promising.

67. Hauser, J. J. and Treuting, R. G.
Relation between structure anisotropy and super-conducting anisotropy. AMERICAN PHYSICAL SOCIETY. BULLETIN 7(4):232. 23 Apr 1962. (abstract)

Cold rolled Mo-40Re and Mo-50Re were found to have a superconducting anisotropy correlated with the structural anisotropy. The ratio of peak to dip increases with the degree of cold rolling. 2 references.

68. Heal, T. J.
The mechanical and physical properties of magnesium and niobium canning materials. In PROCEEDINGS OF THE SECOND UNITED NATIONS INTERNATIONAL CONFERENCE ON THE PEACEFUL USES OF ATOMIC ENERGY HELD IN GENEVA 1 SEPTEMBER–13 SEPTEMBER 1958. VOLUME 5 - PROPERTIES OF REACTOR MATERIALS. Geneva, United Nations, 1958. p. 208–219 (Paper no. 305)

...The properties of niobium and some of its alloys have been studied in relation to their use as canning materials for the Dounreay Fast Reactor. The temperature range for normal operation is low, relative to the melting point, and the niobium cans are required both to contain the fuel and to restrain the growth and distortion under

irradiation. Some work has also been necessary at high temperatures to assess the behavior of the cans under fault conditions. The results reported cover a wide temperature range extending to 1050°C but relate principally to work in the range up to 600°C.

69. Healey, F. H., Chessick, J. J. and Zetlemoyer, A. C.
Adsorption studies on metals. I. Physical
and chemical adsorption of gases on molybdenum.
JOURNAL OF PHYSICAL CHEMISTRY
57:178−182, Feb 1953.

Heterogeneous nature of Mo powder surface, both before and after reduction of surface oxide, was studied by measurements of physical and chemical adsorption of gases. Procedure for reduction and degassing of Mo powder was developed which gives a metal surface apparently free of chemisorbed O_2 and H_2.

70. Hehemann, R. F. and Ault, G. M., eds.
HIGH TEMPERATURE MATERIALS; CONFERENCE
HELD IN CLEVELAND, OHIO, APRIL 16−17, 1957.
N. Y. Wiley, 1959. 556p.

A conference on recent developments in high-temperature materials is presented. In Parts I and II, Ni and Co alloys and cermets are treated. Part III deals with refractory metals.

71. Hein, R. A., Gibson, J. W. and Blaugher, R. D.
Superconductivity of the NbMo alloys at temperatures below 0.25°K. AMERICAN PHYSICAL
SOCIETY. BULLETIN 7(4):322, 23 Apr 1962. (abstract)

The zero-field superconducting transition temperatures of Nb-Mo alloys decrease linearly with molybdenum concentration down to 0.25°K for the Nb-42Mo alloy. Richer molybdenum alloys are superconducting at still lower temperatures. A transition temperature of 0.07°K is indicated for Mo-49Nb. 2 references

72. Hidnert, P. and Gero, W. B.
Thermal expansion of molybdenum. In
U.S. BUREAU OF STANDARDS. SCIENTIFIC
PAPERS. No. 488:429–444, 5 May 1924.

Gives results in figures and tables of expansion tests made for various temperature ranges between room temperature and 750°C on molybdenum ingots prepared from fine and coarse grained molybdenum powders and on samples swaged to various diameters.

73. Hirshfeld, A. T., Leupold, H. A. and Boorse, H. A.
Superconducting and normal specific heats of
niobium. PHYSICAL REVIEW 127:1501–1507,
1 Sep 1962.

NSF-Navy-supported investigation of differences in the published values of the low temperature heat capacity of niobium and of the effect of high-temperature annealing (2,340°C) and degassing on the same sample for which data have previously been published by Brown, Zemansky, and Boorse. The new measurements cover the temperature interval 1.1° to 11.5°K. and are significantly different from those obtained prior to the thermal treatment.

74. Houck, J. A.
PHYSICAL AND MECHANICAL PROPERTIES
OF COMMERCIAL MOLYBDENUM-BASE
ALLOYS. Battelle Memorial Institute,
Columbus, Ohio. DMIC Report 140. 30 Nov 1960.
174p.

The available mechanical and physical properties of unalloyed molybdenum and its commercially available alloys are discussed in this report. Few data on the physical properties of molybdenum alloys exist in the published literature. Because of the small quantities of additions used in current alloys, it can be assumed that the physical properties of the alloys do not differ greatly from those of the unalloyed molybdenum... In addition to the descriptive portions of this report, Appendix A contains tables of the available published data on the mechanical properties of all the alloys that have been considered in this compilation.

75. Howl, D. A. and Davis, A.F.
Emissivities of various niobium surfaces in temperature range 500–1100°C. BRITISH JOURNAL OF APPLIED PHYSICS 13(5):223–226, May 1962.

Specimens were in form of tubes; 5 surfaces were in as-manufactured condition, deoxidized, and 3 types with varying degrees of nitriding; during investigation, it was found that emissitivies altered with heat treatment; sets of results are therefore given for both initial and final conditions, latter being state reached when no further change could be detected.

76. Inouye, H.
SCALING OF COLUMBIUM IN AIR. Oak Ridge National Laboratory, Tenn. ORNL-1565. 24 Sep 1953. 20p.

The scaling of Nb in dry air and in air containing 18.6 mm of water vapor was determined between 400 and 1200°C. In dry air at 400°C, the oxidation proceeds by short periods of rapid oxidation followed by longer periods of slower oxidation, but after an interval of 21 hr, the rate becomes linear. The presence of water vapor greatly accelerated the linear oxidation rate. In dry air at 600°C, the oxidation rate is linear. The presence of moisture in the air reduces the oxidation rate and causes a deviation from linearity. The explanation of this is that an increased surface area results from uneven oxidation when the runs are made in dry air. Above 600°C, the presence of water vapor does not affect the oxidation; the rate is linear. Both black and white oxides were formed at these temperatures and have been identified as Nb_2O_5. Three modifications of the pentoxide were found; the temperatures at which they are stanle are not necessarily those found by Brauer. Rate constants were calculated for each temperature investigated, and an Arrhenius plot was made. A straight line was obtained when the logarithm of the rate constant was plotted vs. 1/T. An inflection of the curve occurs at 900°C, which corresponds to the temperature at which the scale undergoes a modification of its form. Energies of activation were calculated to be 13,400 cal/mole between 900 and 1200°C.

77. Jacobus, H. W.
METALLOGRAPHIC EXAMINATION OF A RIVETED W-2 COATED MOLYBDENUM TEST ASSEMBLY. McDonnell Aircraft Corp., St. Louis, Mo. Report No. 8888. 10 Jul 1962. 10p. [Contract AF 33(657)-7749] ASTIA AD-277 777.

The test box was exposed at 2500°F for one-half hour. It was then exposed to plasma jet temperatures. Holes were drilled prior to plasma jet exposure to determine if the plasma jet environment will initiate coating deterioration. Holes enlarged but no spalling of the coating occurred. The breaks in the W-2 coating occured in sectioning of the specimens. The majority of breaks observed in the coating, and lack of coating continuity, resulted from metallographic preparation. Several areas did have breaks in the coating, however, and loss of base metal through oxidation occurred. Exposure to plasma jet temperatures did not appear to seriously affect the coating when compared to material exposed one-half hour at 2500°F.

78. Jahnke, L. P. and Frank, R. B.
High-temperature metallurgy today. METAL PROGRESS 74(6):86–91, Dec 1958.

Mechanisms for strengthening super alloys and refractory metal compositions for high-temperature service are discussed. Information is presented on:...the advantages and disadvantages of using... molybdenum, columbium,...at elevated temperatures...

79. Jefferys, R. A. and Gadd, J. D.
DEVELOPMENT AND EVALUATION OF HIGH TEMPERATURE PROTECTIVE COATINGS FOR COLUMBIUM ALLOYS. PART II. COATING EVALUATION. Tapco. Div. of Thompson Ramo Wooldridge Inc., Cleveland Ohio. ASD-TR-61-66(Pt. II). Jul 1961. 103p. [Contract AF 33(616)-7215]

An evaluation was made of 18 coating-base metal systems, viz, six different coatings applied to 3 Nb-base materials (D-31 alloy, F-48 alloy and unalloyed Nb). The 18 systems were tested under identical conditions in cyclic oxidation (2300 and 2500°F), thermal shock (2500- to 250°F), bend-oxidation (2500°F), and stress-oxidation (2500°F), plus tensile tests. The tests produced data for the 18 systems relating to the comparative protective nature of each coating, and to the effect of the coating and the coating treatment on the mechanical properties of the substrate.

80. Jones, H. A., Langmuir, I. and Mackay, G.M.J.
The rates of evaporation and the vapor pressures of tungsten, molybdenum, platinum, nickel, iron, copper and silver. PHYSICAL REVIEW 30:201-214, Aug 1927.

Langmuir and Mackay's data on the rates of evaporation of tungsten, molybdenum and platinum have been corrected to the tungsten temperature scale of Forsythe and Worthing. Similar data on nickel, iron, copper and silver are published for the first time on the same temperature scale. The third law of thermodynamics has been assumed in all calculations and the vapor pressures are calculated from the rate of evaporation. The experimental data check the third law as shown by the constancy of the calculated values of λ_0, the latent heat of evaporation at the absolute boiling points. The data are compared with those of other investigators. The evaporation of metals heated in an atomic hydrogen flame was very rapid and gave results which confirmed the data on evaporation in vacuum. The values of λ_0 expressed in cal. gm. (atom)$^{-1}$ and the boiling points in degrees Kelvin are

	W	Mo	Pt	Ni	Fe	Cu	Ag
λ_0	191880	146000	127500	89440	89025	82062	71320
B.P	6970	5960	4800	3650	3475	3110	2740

81. Kannuluik, W. G.
The thermal and electrical conductivities of several metals between -183°C. and 100°C. ROYAL SOCIETY OF LONDON. PROCEEDINGS Ser. A, 141:159-168, 1933.

An electrical method of obtaining the thermal and the electrical conductivity of a metal in wire form, previously developed and used by the author, is extended so as to include observations between -183°C. and 100°C. The wires studied are tungsten (in monocrystal form), molybdenum, silver, and iron. The complete thermal and electrical data obtained are given in Table II. Optical spectroscopic analyses of the purity of the tungsten, molybdenum, and silver wires were made. The nature of the impurities, and in the case of molybdenum and tungsten, their probable amounts are given in Table III of the Appendix.

82. Kessler, H. D. and Hansen, M.
A study of arc-melted molybdenum-rich chromium-molybdenum alloys. AMERICAN SOCIETY FOR METALS. TRANSACTIONS 42:1008–1030, 1950; Disc.: 1030–1032.

Cr-Mo alloys containing 60–100% Mo were arc melted and cast in an argon atmosphere. The melting method consisted in fusing consumable electrodes made by powder-metallurgical techniques. C and Be were investigated as possible deoxidizers. Measured lattice parameters check the results of other investigators, and the body-centered cubic alloys show complete solid solubility. Oxidation resistance in static air was evaluated at 1200, 1500, and 1800°F. Forgeability and hardness were also studied.

83. Kihlborg, L. and Magneli, A.
On the thermal decomposition of molybdenum trioxide in vacuo. ACTA CHEMICA SCANDINAVICA 9(3):471–474, 1955.

MoO_3 partially decomposes at 550–800°C in evacuated silica tubes and forms lower oxides, two of which were previously unknown and were characterized by x-ray diffraction patterns. The compositions and structures of the ξ oxide and two new forms, the ζ and η oxides are not yet known. Data are tabulated.

84. Kingery, W. D. and Norton, F. H.
THE MEASUREMENT OF THERMAL CONDUCTIVITY OF REFRACTORY MATERIALS. Massachusetts Institute of Technology, Cambridge. Quarterly progress report for the period ending 1 Oct 1954. 1954. 7p. [Contract AT(30-1)960]

Additional thermal conductivity data for refractory materials are reported, and new materials are being prepared. Equipment for determining thermal emissivity and studying radiant energy transmission is now being assembled. A furnace for preparing samples of varying crystal size and testing for thermal conductivity and associated effects has been completed.

85. Kirillin, V. A., Sheindlin, A. E. and
Chekhovskii, V. Ya.
Experimental determination of the enthalpy of
molybdenum in the 700°–2337°C range.
AKADEMIIA NAUK SSSR. DOKLADY
139(3):645–647, 21 Jul 1961 (In Russian)

The enthalpy (H_t - H_o, where H_o is enthalpy at 0°C) of molybdenum was determined calorimetrically, using thermocouples up to 2000°C and pyrometers at higher temperatures. Mean values for 32 temperatures were determined. Thus, H_t - H_o = 45.58 kcal/kg at 699.3°C, 139.48 at 1854°C, 190.22 at 2337°C. The maximum relative error was 0.4% when thermocouples were used, 0.9% with pyrometers. 9 references.

86. Kirillin, V. A., Sheindlin, A. E. and
Chekhovskii, V. Ya.
Experimental determination of the enthalpy and
heat capacity of molybdenum up to 2337°C.
INTERNATIONAL JOURNAL OF HEAT AND
MASS TRANSFER 5:1–9, Jan–Feb 1962.

The enthalpy and heat capacity of molybdenum were determined calorimetrically in the 700°–2337°C range. In the previously investigated lower portion of the range, values agreed well. Graphs, tables and diagrams are presented. 10 references.

87. Kling, H. P.
Development of oxidation-resistant niobium
alloys. In TECHNOLOGY OF COLUMBIUM
(NIOBIUM). N.Y., Wiley, 1958. p. 87–91.

Samples of Cb and Cb alloyed with V, Mo, or Ti were oxidized in air at elevated temperatures. Results of microscopic, chemical and electrical conductivity studies on oxide scale form basis for discussion of oxidation rate and mechanism.

88. Klopp, W. D., Sims, C. T. and Jaffee, R. I.
EFFECTS OF ALLOYING ON THE KINETICS OF OXIDATION OF NIOBIUM. Geneva, Second United Nations Conference on the Peaceful Uses of Atomic Energy, 1958. Report A/CONF.15/P/712. 39p.

Kinetics of the Cb-0 and Cb-air reactions investigated at 400−550°C. and 600−1200°C. respectively. Effects of binary additions of V, Mo, Cr, Ti, Zr and W on the Cb-Air reaction investigated at 1000 and 1200°C. Weight-grain data were obtained for Cb ternary alloys in air at 600, 800 and 1000°C.

89. Klopp, W. D., Sims, C. T. and Jaffee, R. I.
High temperature oxidation and contamination of columbium. AMERICAN SOCIETY FOR METALS. TRANSACTIONS 51:282−298, 1959.

The oxidation and contamination rates of columbium in oxygen and in air were measured in the temperature range 600 and 1200°C (1110 and 2190°F). The oxidation curves in oxygen were linear and the rate constants were correlated by an Arrhenius-type plot. The heat of reaction was 5410 cal per mole from 600 to 1100°C (1110 to 2010°F); above 1100°C (2010°F) the reactions were rapid and the rates could not be correlated to the Arrhenius-type plot. The activation energy for diffusion of oxygen in columbium was calculated from contamination data. Weight-change curves of columbium in air were also linear, with lower rates than in oxygen. The heat of reaction was, from the reaction rate data, 10,100 cal per mole from 600 to 1200°C (1110 to 2190°F). Contamination in air-reacted columbium was similar to that in oxygen-reacted columbium, suggesting that oxygen is the primary diffusing contaminant.

90. Klopp, W. D., Berry, W. E. and Maykuth, D. J.
The hot water corrosion resistance of columbium and columbium alloys. In COLUMBIUM METALLURGY; PROCEEDINGS OF A SYMPOSIUM SPONSORED BY THE HUDSON-MOHAWK SECTION, IN COOPERATION WITH THE METALLURGICAL SOCIETY, AMERICAN INSTITUTE OF MINING, METALLURGICAL, AND PETROLEUM ENGINEERS, BOLTON LANDING, NEW YORK, JUNE 9–10, 1960. Douglass, D. L. and Kunz, F. W., eds. New York, Interscience, 1961. p. 685–718 (Metallurgical Conferences, v. 10)

Corrosion studies were conducted in 600 and 680°F water and in 750°F steam on a large number of wrought binary and ternary columbium alloys. These were prepared using either commercial or high purity columbium as the base material. High purity columbium shows a greater degree of corrosion resistance than the commercial grade. However, base-metal purity does not appear to have a significant effect on the corrosion behavior of columbium alloys. While many alloying additions improve the corrosion resistance of columbium, the effectiveness of binary titanium, zirconium, and vanadium additions of ternary titanium and chromium combinations is outstanding. The corrosion studies were supplemented with metallographic and x-ray diffraction studies. The hot water corrosion resistance of these alloys was correlated with their resistance to oxidation in air at elevated temperatures.

91. Klopp, W. D. et al
OXIDATION AND CONTAMINATION REACTIONS OF NIOBIUM AND NIOBIUM ALLOYS. Battelle Memorial Institute, Columbus, Ohio. BMI-1317. 3 Feb 1959. 62p.

Cb reacts linearly with air at 600 to 1200°C. and with oxygen at 400 to 1400°C. At 1400°C a very rapid reaction with oxygen is observed. Contamination hardening during oxidation is attributed to diffusion of oxygen into the metal from the predominantly Cb_2O_5 scale. The most effective binary additions for improving oxidation resistance are Cr, Mo, Ti and V.

92. Klopp, W. D.
OXIDATION BEHAVIOR AND PROTECTIVE COATINGS FOR COLUMBIUM AND COLUMBIUM-BASE ALLOYS. Battelle Memorial Institute, Columbus, Ohio. DMIC Report No. 123
15 Jan 1960. 98p.

An adherent scale first forms and follows parabolic behavior. This is followed by a tarnish film which blisters and has a tendency to sinter at 900°C. The high solubility and high diffusion rate of oxygen in Cb causes contamination and embrittlement. Nitrogen does not affect these reactions. Binary and ternary alloys of Cr, Mo, Ti, W, V and Zr inhibit oxidation. Coatings of LM5 and a modified Ni-Cr alloy are reasonably effective.

93. Kofstad, P. and Kjollesdal, H.
Oxidation of niobium (columbium) in the temperature range 500° to 1200°C. AIME. METALLURGICAL SOCIETY. TRANSACTIONS
221:285-294, Apr 1961.

Kinetic studies of the oxidation and structural investigations of the oxidized specimens by x-ray and electron diffraction, electron microscopy and metallographic techniques at 500-1200°C and 0.1-760 mm. of Hg. The oxidation reaction has a highly irregular temperature dependence and is unusually sensitive to changes in the oxygen pressure.

94. Kolski, T. L. and Hicks, W. T.
Columbium-oxygen reactions from 450 to 1200°C. JOURNAL OF METALS
11:577, 1959. (abstract)

The reaction rate of pure columbium with dry oxygen (1 atm) was measured to 50 pct of completion by a continuous weight gain technique in the temperature region 450 to 1200°C. These measurements show few regions of truly linear oxidation implied by some earlier workers but do confirm the transition from semi-protective to non-protective oxidation and the rate reversal phenomenon reported previously. Motion picture studies of the reaction clarify the anomalies by revealing a physical change in the nature of the oxide formed as a function of time and temperature. However, x-ray diffraction studies indicate that the same phase of Cb_2O_5 is formed throughout the temperature region of the anomalies. Continuous weight gain measurements on CbO are also reported and some tentative mechanisms for the oxidation of columbium are suggested.

95. Krikorian, O. H.
HIGH-TEMPERATURE STUDIES: I. REACTIONS OF REFRACTORY SILICIDES WITH CARBON AND NITROGEN. II. THERMODYNAMIC PROPERTIES OF THE CARBIDES. III. HEAT OF FORMATION OF THE $3\pi_u$ STATE OF C_2 FROM GRAPHITE. California. Univ. Radiation Laboratory, Livermore. Report UCRL-2888. 1955. 136p. [Contract W-7405-Eng-48] ASTIA AD-63 120.

The reactions of silicides of ...Nb,...Mo...with carbon and with nitrogen were studied at temperatures around 2000°K. The data were used to establish ternary phase diagrams and to set upper and lower limits on the heats of formation of the silicides. Methods are suggested for estimating absolute entropies of carbides. The heat of formation of the $3\pi_u$ state of C_2 has been determined as 191.4 ± kcal at 0°K.

96. Krikorian, O. H.
THERMAL EXPANSION OF HIGH TEMPERATURE MATERIALS. California. Univ., Livermore. Lawrence Radiation Laboratory. Report UCRL-6132 6 Sep 1960. 7p. [Contract W-7405-eng-48].

Thermal expansion data, presented as per cent expansion from 25°C to 500°, 1000°, 1500°, 2000° and 2500°C, are tabulated for ...molybdenum...$MoSi_2$...

97. Krockel, O.
A new thermo-element for high temperature measurements. SILIKAT-TECHNIK 11(3):108–111, Mar 1960 (In German)

A discussion is presented of a $MoSi_2$-Mo thermoelement. Topics considered include production of the element, its physical properties, and applications. Structural micrographs are presented and data are shown in graphs.

98. **Kubaschewski, O. and Evans, E. Ll.**
METALLURGICAL THERMOCHEMISTRY.
N.Y., Pergamon, 1951. 368p.

Opens with the elementary account of the main thermodynamic functions used in the classical treatment of solutions and heterogeneous equilibria. Experimental techniques commonly used to obtain thermal, free-energy, and equilibrium data. Methods of estimating thermochemical data, in order that approximate calculations may be made of the thermodynamic properties of systems for which complete experimental data are not available. Thermodynamic properties for many elements and compounds of metallurgical interest. Examples of the thermodynamic treatment of metallurgical problems.

99. Kubaschewski, O. and Hopkins, B. E.
OXIDATION OF METALS AND ALLOYS.
N.Y., Academic Press, 1953. 239p.

After background information on oxidation, there are sections on experimental methods, special theories concerning different types of oxidation, and experimental results relating to the rate of oxidation of various metals and alloys.

100. Latva, J. D.
Selection and fabrication of nonmetallics – oxides, beryllides and silicides. METAL PROGRESS 82(5):97–102,124,126,128, Nov 1962.

Oxides, beryllides and silicides will be used more for structural components as our understanding of brittle fracture increases and better processing methods are developed. Applications are growing as engineers recognize the unique combinations of properties which are possible – chemical and thermal stability, low thermal and electrical conductivity and high strength-to-density ratios.

101. Lazareva, L. S., Kantor, P. B. and Kandyba, V. V.
Enthalpy and thermal capacity of molybdenum in the temperature range 1200° to 2500°K. PHYSICS OF METALS AND METALLOGRAPHY 11(4):133–134, Apr 1961.

The enthalpy of molybdenum was determined in the 1164°–2540°K range. Results are tabulated, as are heat capacities calculated from them. 7 references.

102. Lehman, G. W.
THERMAL PROPERTIES OF REFRACTORY MATERIALS. Atomics International Div., of North American Aviation Inc., Canoga Park, Calif. WADD-TR-60-581. Jul 1960. 19p.
[Contract AF33(616)-6794]

A pulse heating method for measuring the specific heat of conductors from near absolute zero to their melting point is described. Results obtained on copper, iron, molybdenum, tantalum, and rhenium are reported. They indicate that the pulsed heating method gives specific heat data accurate to a few percent. The measurements show that the heat capacities of molybdenum and tantalum gradually rise above their Dulong and Petit values of 0.06253 and 0.03316 cal/gm/°C, respectively, at high temperatures. At 2800°K, c_p (Mo) = 0.1350 cal/gm/°C and at 3200°K, c_p (Ta) = 0.0667 cal/gm/°C. These anomalies were noted in other transition metals. The percentage elongation, relative to room temperature, of zirconium carbide was measured between 1000 and 2000°C and was found to vary linearly from 0.6% at 1000°C to 1.6% at 2300°C with a permanent set occurring above 2300°C. A transient method for measuring thermal diffusivity, specific heat, and thermal conductivity of insulators as well as conductor is under development for the temperature range 20°C to 2500°C.

103. Long, R. A.
FABRICATION AND PROPERTIES OF HOT-PRESSED MOLYBDENUM DISILICIDE.
National Advisory Committee for Aeronautics,
Washington, D. C. NACA RM E50F22, 1950, 34p.

Hot-pressed molybdenum dislicide bodies produced by industrial methods and sources were evaluated. The outstanding characteristics reported are an elevated-temperature (2400°) short-time tensile strength of 41,070 psi and a 100-hour rupture stress at 1800°F. in excess of 30,000 psi as indicated in a preliminary evaluation. The material evaluated was extremely resistant to air corrosion up to 2850°F and generally brittle at temperatures below 2200°F.

104. Lustman, F. B.
Oxidation of molybdenum in air at 1100° to 1600°F. METAL PROGRESS
57:629– 630, 674, May 1950.

Fundamental principles and actual behavior of Mo and its oxides at above temperatures in air. Experimental data on oxide production on heating for various times and at different temperatures. Amount of oxide which was strongly adherent was also determined. Indicates principles that will lead to a Mo-base alloy with a nonvolatile, impervious, refractory, and protective scale.

105. McIntosh, A. B.
Development of niobium. INSTITUTE OF METALS. JOURNAL 85:367–382,
Apr 1957.

Columbium's properties suggest use as canning material for nuclear reactors; data on chemical, physical and mechanical properties indicate potentialities of use as structural materials for liquid metal, heat transfer units or chemical processing equipment, electrical equipment; discussion of binary columbium alloy systems.

106. Marden, J. W. and Wroughten, D. M.
The effect of working on the physical properties of molybdenum. ELECTROCHEMICAL SOCIETY. TRANSACTIONS 89:216, 1946.(abstract)

Values are recorded for hardness, ultimate tensile strength, and percentage elongation of Mo wire and sheet having had various amounts of mechanical working. The data indicate only the range that may be expected and are not exact for any given piece of metal. The methods of working, annealing, etc., greatly affect the strength and ductility of the metal. Mo metal is inherently a moderately hard, very brittle metal and the properties of high strength and ductility found in commercial metal are properties imparted to it by mechanical and heat-treatment.

107. Martin, M. J.
The photoelectric and thermionic properties of molybdenum. PHYSICAL REVIEW 33:991–997, Jun 1929.

The thermionic emission as a function of temperature was plotted logarithmically according to Richardson's equation. The value of the thermionic work function determined from the slopes of these curves was 3.48 ± .07 volts. The photoelectric and thermionic work functions were thus found to agree within 0.26 volts.

108. No Abstract

109. Mauer, F. A. and Bolz, L. H.
MEASUREMENT OF THERMAL EXPANSION OF CERMET COMPONENTS BY HIGH TEMPERATURE X-RAY DIFFRACTION. U.S. National Bureau of Standards, Washington, D. C. NBS Report No. 5837; suppl. to WADC Technical Report no. 55-473. 1957. 47p. [Contract AF33(616)-56-5] ASTIA AD-155 555.

110. Mauer, F. A. and Bolz, L. H.
MEASUREMENT OF THERMAL EXPANSION OF CERMET COMPONENTS OF HIGH TEMPERATURE: X-RAY DIFFRACTION. U.S. National Bureau of Standards, Washington, D.C. Summary report for 14 Feb 1954 – 14 Sep 1955. 1955, 63p. [Contract AF33(616)-53-12] (WADC-TR-55-473) ASTIA AD-95 329.

A high-temperature specimen mount for a commercial x-ray-diffraction unit was designed especially for accurate measurements at temperatures up to 1400°C. It has been used in making thermal expansion measurements on seventeen samples, most of which are refractory materials being considered for use in high temperature applications. In general, the precision attained is comparable to that attained with conventional dilatometers but the x-ray method is unique in that it can provide data on the expansion in different directions in anisotropic crystals when only a few grams of powdered material are available. There is a further advantage in that the expansion measured is that of the crystal lattice and is independent of effects such as hysteresis that are observed in polycrystalline bodies because of changes in the way the crystallites fit together.

111. Mauer, F. A. and Bolz, L. H.
THERMAL EXPANSION OF CERMET COMPONENTS BY HIGH TEMPERATURE X-RAY DIFFRACTION. National Bureau of Standards, Washington, D. C. Progress Report No. 7. NBS Report No. 3824. 1954. 16p. [Contract AF33(616)53-12]

112. Maxwell, W. A.
OXIDATION-RESISTANCE MECHANISM AND OTHER PROPERTIES OF MOLYBDENUM DISILICIDE. National Advisory Committee for Aeronautics, Washington, D.C. NACA RM E52A04. 1952. 17p.

An experimental investigation was conducted to determine the oxidation-resistance mechanism, the melting characteristics, the electric resistivity, and the modulus of elasticity of molybdenum disilicide. Molybdenum disilicide $MoSi_2$ bodies are protected from oxidation at high temperatures by the formation of a protective siliceous coating in which alpha-cristobalite has been identified as the sole or principal constituent. In powered form, $MoSi_2$ is not inherently resistant to oxidation and burns in air at comparatively low temperatures. Molybdenum disilicide decomposed at the melting point with the loss of appreciable silicon. This phenomenon has so far prevented casting of satisfactory ingots. Decomposition below the melting point was indicated. The electric resistivity of $MoSi_2$ at room temperature varies from 23 to 27 microhm-centimeters depending on the method of fabrication. The conductivity was found to be metallic in nature and to decrease with increasing temperatures to 200°F. The room-temperature modulus of elasticity as determined by a dynamic method was 59X10° pounds per sq. in. Poor damping characteristics are indicated for the material at room temperature.

113. Maxwell, W. A.
PRELIMINARY INVESTIGATION OF PLATE-TYPE MOLYBDENUM DISILICIDE FUEL ELEMENTS FOR AN AIR-CYCLE NUCLEAR REACTOR. National Advisory Committee for Aeronautics, Washington, D.C. NACA Research Memorandum E52L18. 1953. 11p.

Flat, plate-type elements 3.5 x 0.5 x 0.070 in., containing 10% elemental natural uranium, were produced by hot pressing. The elements were acid-treated to remove surface uranium and then coated with an alumina glaze. Such elements remained stable and did not lose detectable quantities of fission fragments in a 400-hr treatment at 1800°F in a flux of 5×10^{11} neutrons/cm^2/sec. They are conviently produced and, dependent on design and on the successful resolution of problems such as thermal shock, offer a possible method of increasing air-cycle element temperatures and related performance.

112. Maxwell, W. A.
OXIDATION-RESISTANCE MECHANISM AND OTHER PROPERTIES OF MOLYBDENUM DISILICIDE. National Advisory Committee for Aeronautics, Washington, D. C. NACA RM E52A04. 1952. 17p.

An experimental investigation was conducted to determine the oxidation-resistance mechanism, the melting characteristics, the electric resistivity, and the modulus of elasticity of molybdenum disilicide. Molybdenum disilicide $MoSi_2$ bodies are protected from oxidation at high temperatures by the formation of a protective siliceous coating in which alpha-cristobalite has been identified as the sole or principal constituent. In powered form, $MoSi_2$ is not inherently resistant to oxidation and burns in air at comparatively low temperatures. Molybdenum disilicide decomposed at the melting point with the loss of appreciable silicon. This phenomenon has so far prevented casting of satisfactory ingots. Decomposition below the melting point was indicated. The electric resistivity of $MoSi_2$ at room temperature varies from 23 to 27 microhm-centimeters depending on the method of fabrication. The conductivity was found to be metallic in nature and to decrease with increasing temperatures to 200°F. The room-temperature modulus of elasticity as determined by a dynamic method was 59X10° pounds per sq. in. Poor damping characteristics are indicated for the material at room temperature.

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114. Maxwell, W. A.
PROPERTIES OF CERTAIN INTERMETALLICS AS RELATED TO ELEVATED-TEMPERATURE APPLICATIONS. I. MOLYBDENUM DISILICIDE. National Advisory Committee for Aeronautics. Washington, D.C. NACA RM E9G01. 1949, 27p.

115. Maykuth, D. J. et al
DEVELOPMENT OF CORROSION RESISTANT NIOBIUM-BASE ALLOYS. Battelle Memorial Institute, Columbus, Ohio. BMI-1437, 12 May 1960. 90p.

The hot water corrosion resistance and mechanical properties of 99.8% Cb and a number of its alloys are evaluated to determine their usefulness in pressurized water thermal reactors. Cr, Mo, Ti, V, Zr, W, Al, Fe, Ni, Ce and Pd are chief alloying elements.

116. Maykuth, D. J. and Jaffee, R. I.
The fabrication and mechanical properties of some columbium alloys for use in pressurized water reactors. In COLUMBIUM METALLURGY; PROCEEDINGS OF A SYMPOSIUM SPONSORED BY THE HUDSON-MOHAWK SECTION, IN COOPERATION WITH THE METALLURGICAL SOCIETY, AMERICAN INSTITUTE OF MINING, METALLURGICAL, AND PETROLEUM ENGINEERS, BOLTON LANDING, NEW YORK, JUNE 9–10, 1960. Douglass, D. L. and Kunz, F. W., eds., New York, Interscience, 1961. p. 223–256. (Metallurgical Conference, v. 10)

A number of binary and ternary alloys of interest in an alloy development program for pressurized-water reactor applications were prepared, using an electron beam refined columbium base, by arc melting and fabricated to sheet. The comparative fabricabilities of these alloys and their hardnesses, tensile, creep, and stress-rupture properties at temperatures up through 1500°F are discussed.

117. Mezaki, R., et al
HIGH TEMPERATURE HEAT CONTENTS OF FIFTEEN REFRACTORY BORIDES. Preprint SM-26/48. 27p. (In English) (To be published in the PROCEEDINGS OF THE IAEA SYMPOSIUM ON THE THERMODYNAMICS OF NUCLEAR MATERIALS held in Vienna, 21–25 May 1962.)

High precision thermodynamic properties for fifteen refractory borides (BN (hexagonal), BN(cubic), TiB_2, ZrB_2, HfB_2, TaB, TaB_2, CrB, CrB_2, Mo_2B, MoB, MoB_2, W_2B, WB, and W_2B_5) were determined with a copper-block drop-type calorimeter over the range 400 to 1200°K. Gold containers were used in all the experiments to minimize side reactions. From a consideration of available low temperature measurements and a new technique for fitting the high temperature data with Debye functions, it was possible to estimate S^0_{298} for these substances with a reliability of ± 1 entropy unit.

118. Michael, A. B.
The oxidation of columbium-base and tantalum-base alloys. In REACTIVE METALS; PROCEEDINGS OF THE THIRD ANNUAL CONFERENCE SPONSORED BY NIAGARA FRONTIER SECTION IN COOPERATION WITH THE METALLURGICAL SOCIETY, AMERICAN INSTITUTE OF MINING, METALLURGICAL, AND PETROLEUM ENGINEERS, BUFFALO, NEW YORK, MAY 27–29 1958. Clough, W. R., ed. New York, Interscience, 1959. p. 487–507. (METALLURGICAL SOCIETY CONFERENCES, v. 2)

The oxidation of arc-melted columbium, tantalum and alloys of these metals with Ti, Zr, W, Mo, V, Cr, Co, Ni, Fe, Si, and Al was investigated. Alloys were surveyed by exposure to flowing air at 2000°F for 16 hr and selected compositions were investigated for other temperatures and times. The highest resistance to scaling were observed for Cb-Cr-Co, Cb-Ta-Cr-Co and Ta-Cr-Co alloys. For these alloys, lower resistance to scaling were observed when Fe was substituted for Cr and Ni for Co. Other alloys which had significant resistances to scaling at 2000°F were:...Cb-Mo... in the order of decreasing resistances.

119. Molybdenum.
METALEN 16(21):363–366, 15 Jan 1962
(In Dutch)

Mechanical and physical properties, resistance to corrosion, and workability of molybdenum are briefly reviewed. The mechanical properties, which are almost the same in arc cast and sintered molybdenum, vary considerably with the degree of plastic deformation (rolling). Another important factor is the position of the transformation point of molybdenum (in the 300°–400°C range), which depends on the plastic and thermal pretreatment of the material. When quenched from a temperature above the transformation point, the metal is brittle at room temperature. 1 reference.

120. Molybdenum alloy as high melting point.
MATERIALS IN DESIGN ENGINEERING
52(2):202,204, Aug 1960.

Climax Molybdenum Co., has now made available its Mo-30W alloy, Climelt, which has a melting point of 5100°F and a theoretical density of 0.43 lbs/in^3. Intended for high temperature service, the alloy has been successfully forged at 2300°F, although more reheating and higher working pressures are required than for molybdenum. Conventional or spark-discharge machining may be used.

121. Morris, J. C., Schurin, B. and Olson, O. H.
The total emissivity of aircraft structural materials. In THERMODYNAMIC AND TRANSPORT PROPERTIES OF GASES, LIQUIDS AND SOLIDS. Touloukian, Y. S., ed. N.Y., McGraw-Hill, 1959. p. 400–404.

The total normal emissivities of pure molybdenum and other metals were measured bolometrically as a function of temperature over the range -320°-2500°F. Each sample was cycled three times. Data tabulated.

122. Nachtigall, E.
Properties of molybdenum and tungsten at low and medium temperatures. ZEITSCHRIFT FUER METALLKUNDE. 43:23–26, Jan 1952. (In German); POWDER METALLURGY BULLETIN 6:26–31, Apr 1951

Oxidation behavior, hardness, tensile strength, and elongation of Mo and W below 500°C.

123. National Research Council. Materials Advisory Board, Washington, D. C.
SUMMARY REPORT OF THE COMMITTEE ON REFRACTORY METALS. VOLUME I. SUMMARY Report No. MAB-154-M(Vol. I). 15 Oct 1959. 40p. [Contract DA-36-039-sc-76436]

A study was made of the refractory metals, Mo, Nb, W, Ta, Cr, V, Re, and the Pt group to identify the metals which offer the most attractive properties for service at temperatures above 2000°F in missiles and high-speed aircraft. The Refractory Metals Committee concluded that the most important metals are Mo, Nb, W, and Ta.

124. Neel, D. S. and Pears, C. D.
High temperature thermal property measurement to 5000°F. In PROGRESS IN INTERNATIONAL RESEARCH OF THERMODYNAMIC AND TRANSPORT PROPERTIES; SECOND SYMPOSIUM ON THERMOPHYSICAL PROPERTIES, JANUARY 24–26, 1962, PRINCETON UNIVERSITY, PRINCETON, NEW JERSEY. Masi, J. F. and Tsai, D. H., eds. New York, American Society of Mechanical Engineers, 1962. p. 500–511.

The thermal expansion, thermal conductivity, and heat capacity of several refractory materials were determined from room temperature to 5000°F or to the destruction

point of the material. High temperature furnaces with helical graphite heaters were used for heating the specimens. Thermal expansion measurements were made with graphite dilatometers, thermal conductivity measurements were made with a miniaturized radial heat flow apparatus, and heat capacity measurements with a drop-type ice calorimeter. Specimen temperatures were measured by means of optical pyrometers and high temperature thermocouples. The thermal properties were determined for graphite, tungsten, several metallic alloys, and several carbides, nitrides, borides, and oxides. Quite often, the materials failed at temperatures below the expected melting point. The thermal property values varied considerably with temperature on most materials, and contained inflections that often could be explained by changes in structure; however, inflections that could not be anticipated were rather common.

125. Neshpor, V. S. and Samsonov, G. V.
Electrical, thermoelectric and galvanomagnetic properties of silicides and transition metals.
SOVIET PHYSICS – DOKLADY 5(4):877–880, Jan–Feb 1961.

Electrical resistivity, thermoelectric emf and Hall constant of silicides of metals belonging to Groups IV-VI of the periodic table are discussed. Two series of silicides could be distinguished. One series, composed of silicides of ... molybdenum... is characterized by a resistivity which rises monotonically with the silicon content in the molecule and which, in the higher silicides, has a positive temperature coefficient (metallic resistivity). The other series has opposite characteristics (semiconductor resistivity).

126. Neshpor, V. S. and Samsonov, G. V.
Investigation of the Hall effect in silicides of the transition metals. SOVIET PHYSICS – DOKLADY 5(5):1097–1098, Mar–Apr 1961.

Coefficients of the Hall effects were measured in silicides of ... molybdenum (Mo_3Si, Mo_5Si_3, $MoSi_2$), and other transition metals. The results were used for calculating effective concentrations of the current carriers and their Hall mobilities. The relationships observed are of a qualitative character, and they reflect semiconductive phenomena involved, relative intensities of covalent and metallic bonds, etc. In silicides of molybdenum the effect concentration of current carried decreases, and their mobility decreases, with increasing x/y in Mo_xSi.

127. Neshpor, V. S. and Samsonov, G. V.
Study of the electrical conductivity of silicides of the transition metals. SOVIET PHYSICS-SOLID STATE 2(9):1966–1972, Mar 1961.

Data are tabulated for the specific electrical resistivity of ...Mo_3Si, Mo_5Si_3, $MoSi_2$, $MoSi_{1.6}Al_{0.4}$... and other transition metal silicides. The silicides noted are metallic conductors. Electrical resistivities are related to the acceptor capacity of the d-shell of the metal atoms. The temperature coefficient of electrical resistivity in $MoSi_{1.6}Al_{0.4}$ is greater than for binary silicides.

128. Northcott, L.
MOLYBDENUM. New York, Academic, 1956. 222p. (Metallurgy of the Rarer Metals v.5)

The coverage of this work includes:...physical properties of molybdenum...

129. Paprocki, S. J. and Stacy, J. T.
INVESTIGATION OF SOME NIOBIUM-BASE ALLOYS. Battelle Memorial Institute, Columbus, Ohio. Report BMI-1143. 31 Oct 1956. 43p. [Contract W-7405-eng-92]

Twenty-seven Nb-base alloys, containing 5 wt.% of ...Mo...and with an addition of 0, 9.5, or 19.0 wt.% Ti, were examined for workability, ductility, hardness at room temperature and 1600°F, and oxidation resistance at 1800°F. Addition of Ti generally improved workability, increased hot hardness, and promoted oxidation resistance. Some exceptions to this pattern were noted: the hardest alloy at 1600°F, Nb wt.% Zr, softened with added Ti, and the oxidation resistance Nb wt.% Mo and the Nb wt.% V alloys was not increased by additions of Ti.

130. Parke, R. M.
Molybdenum: a new high-temperature metal.
METAL PROGRESS 60:81–96, Jul 1951.

A new role for Mo – its use as a structural material resistant to the damaging effects of temperature. Various physical and mechanical properties related to its new uses.

131. Pears, C. D.
The determination of the emittance of refractory materials to 500°F. In PROGRESS IN INTERNATIONAL RESEARCH ON THERMODYNAMIC AND TRANSPORT PROPERTIES; SECOND SYMPOSIUM ON THERMO-PHYSICAL PROPERTIES, JANUARY 24–26, 1962, PRINCETON UNIVERSITY, PRINCETON, NEW JERSEY. Masi, J. F. and Tsai, D. H., eds. New York, American Society of Mechanical Engineers, 1962. p. 588–598.

The emittances of various refractory materials were determined at temperatures from 800 to 5000°F. The measurements were made by comparing the irradiance from a sample of the material to that from a black body cavity maintained at the same temperature. A thermopile-type detector was employed. Small disc-shaped specimens were heated in an inert atmosphere by lacing them on a disc that was inductively heated by a flat induction coil. The specimen temperature was measured by means of surface thermocouples and an optical pyrometer. The optical pyrometer readings were corrected to true temperatures for the measure emittance with the aid of thermocouple calibration, gray body assumption, and spectral measurements. The emittances of ... molybdenum.. and silicides were determined to their destruction temperatures... The emittances of the different materials varied considerably with temperature and showed many inflections which usually could be associated with material phase changes and expansion coefficients, but which quite often could not be predicted.

132. Pears, C. D. and Neel, D. S.
Performance of refractory materials to 5000°F.
CERAMIC AGE 76(5):30–35, Nov 1960.

Evaluation of such refractory materials as molybdenum... and nitrides and diborides of these metals is described. Thermal and electrical properties were measured and mechanical property data was refined by Southern Research Institute. Melting point data are tabulated and thermal conductivity, thermal expansion and heat capacity curves are shown.

133. Peart, R. F., Graham, D. and Tomlin, D. H.
Tracer diffusion in niobium and molybdenum.
ACTA METALLURGICA 10(5)519-523, May 1962.

Radioactive tracer methods were used to study diffusion in molybdenum up to 2330°C. Grain boundary diffusion is the predominant feature even as high as 2000°C. Lattice diffusion within individual grains was measured, and differences with other work were observed. Micrographs are shown. 8 references.

134. Peckner, D.
Columbium, a bright future, but how soon?
MATERIALS IN DESIGN ENGINEERING
54:107-116, Dec 1961.

The status and future of niobium metal and alloys are discussed by comparison with molybdenum, tantalum and tungsten. Particular emphasis is placed on the application, fabrication, properties and weldability of the alloys.

135. Point defects in niobium, molybdenum and tantalum. NATURE 195(4837):169, 14 Jul 1962.

Irradiation of Cb and Ta wire at 40°C. for 3, 6, 9 and 12 months and plastic deformation at room temperature under strain. Determination of recovery peaks, isochronal and isothermal recovery curves, activation energy, electrical resistance and lattice vacancies.

136. Potter, H. H.
Electrical resistance and thermo-electric power of the transition metals. PHYSICAL SOCIETY. PROCEEDINGS 53:695-705, Nov 1941.

The resistances and thermo-electric powers of the transition metals and their variations with temperature have been measured. The relation of the results to the theory of Mott and Jones is discussed.

137. Properties of materials: columbium, tantalum, tungsten, molybdenum, beryllium-wrought. MATERIALS IN DESIGN ENGINEERING 50(5):140. Mid-Oct 1959 (Materials Selector Issue)

Properties tabulated for molybdenum include: physical properties (density, melting point, thermal conductivity, thermal expansion coefficient, specific heat, electrical resistivity);

138. Rasor, H. S. and McClelland, J. D. Thermal properties of graphite, molybdenum and tantalum to their destruction temperatures. JOURNAL OF THE PHYSICS AND CHEMISTRY OF SOLIDS 15(1/2):17-26, Aug 1960.

Thermal expansion specific heat and thermal conductivity were determined for molybdenum from 1000° to 2620°C, and the results are considered analytically. No unusual features were noted (except those due to impurity evolution) in thermal expansion and conductivity behavior. Specific heat exceeded the Dulong-Petit value by a considerable amount. Graphs are shown.

139. Rawson, J. D. W. and Regart, J. McG. Comparison of tensile properties of zone-refined niobium rod and sheet. INSTITUTE OF METALS. JOURNAL 90(11):448 Jul 1962.

Lower yield and ultimate tensile stress, elongation and Vickers hardness values are determined at room temperature for specimens vacuum annealed at 1100°C and given equal reduction in area to determine effects of different methods of fabrication on mechanical properties.

140. Rengstorff, G. W. P.
Search for oxidation resistant alloys of molybdenum
JOURNAL OF METALS 8:171–176
Feb 1956.

In an effort to find an oxidation-resistant alloy of molybdenum, binary and ternary alloys containing aluminum, chromium, cobalt, iron, nickel, silicon, titanium, tungsten, vanadium and zirconium were screened. Fourteen other alloying additions also tested. Many of the alloys were more oxidation-resistant than molybdenum, but none were entirely satisfactory.

141. Rowe, G. W.
Coatings on molybdenum for high temperature sliding. SCIENTIFIC LUBRICATION
11(10):12–15, Oct 1959.

The friction of various molybdenum coatings in sliding contact at high temperatures was studied and compared with that of uncoated molybdenum. Experiments were conducted in vacuum. The friction of molybdenum to molybdenum contact increases with temperature, seizing becoming complete at 1100°C. Allowing H_2S into the vacuum apparatus permitted formation of MoS_2 and gave the lowest friction. Lubrication is maintained in vacuum up to 800°C. MoS_2 coatings are not suitable for use in air. $MoSi_2$ coatings have a higher friction than MoS_2 but permit sliding in air. Graphs are shown.

142. Rudkin, R. L.
THERMAL PROPERTIES OF MOLYBDENUM BETWEEN 2700°R and 4000°R. U.S. Naval Radiological Defense Laboratory, San Francisco, Calif. Technical Report no. 433. 20 Jun 1960. 23p.

Thermal properties of molybdenum were measured in the 2700°–4000°R range. Thermal conductivity was linear. Values were obtained for heat capacity and total hemispherical emittance.

140. Rengstorff, G. W. P.

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142. Rudkin, R. L.

THERMAL PROPERTIES OF MOLYBDENUM

BETWEEN 2700°R and 4000°R. U.S. Naval

Radiological Defense Laboratory, San Francisco,

Calif. Technical Report no. 433. 20 Jun 1960. 23p.

Thermal properties of molybdenum were measured in the 2700°–4000°R range. Thermal conductivity was linear. Values were obtained for heat capacity and total hemispherical emittance.

143. Samsonov, G. V. and Portnoy, K. I.
ALLOYS BASED ON HIGH MELTING COMPOUNDS.
(Splavy na Osnove Tugoplavkikh Soedineniy)
Moscow, State Scientific-Technical Publishing
House of Defense Literature, 1961. 387p.
(English trans. FTD-TT-62-430) (Available from
Office of Technical Services, Washington 25, D. C.)

Means of identifying alloys based on high-melting compounds, their physicomechanical and mechanical properties, and techniques for producing them are presented. Brief descriptions are given of binary and ternary systems formed between high melting metals (...Nb...Mo...) and ...silicon, reciprical alloys of ...silicides, and alloys based on non-metallic high-melting compounds. Examples of industrial applications of the alloys are also given. Coatings formed by high-melting compounds are also described.

144. Samsonov, G. V., Sinel'nikova, V.S. and Kislin, P. O.
Alloys of the system boron-carbide-molybdenum
disilicide. AKADEMIIA NAUK UKRAINS'KOI R.S.R.
DOPOVIDI 8:866–868, 1959 (In Ukrainian)

B_4G-MoS_2 alloys combine heat resistance with high values of thermoelectromotive force, a combination which makes these materials suitable for high-temperature thermocouples. However, at some proportions of the components, the material is readily oxidizible, and, since its thermoelectric properties are also a function of the composition, the necessity of a closer study was recognized. The authors observed the formation of a very hard phase $Mo_x(Si,B,S)_y$ of a wide homogeneity range, which becomes the material of a monophase state at 1:1 ratio of the components. At this point the oxidizing effect is at a minimum and practically zero. On the other hand, the thermoelectromotive force decreases with the increase of MoS_2 content from 10 to 50%.

145. Samsonov, G. V. and Verkhoglyadova, T. S.
The physical properties of transition metal nitrides. AKADEMIIA NAUK SSSR. DOKLADY 142:608–611, 21 Jan 1962 (In Russian)

Values of the thermoelectric coefficient (μv/deg), the electrical resistance, the Hall coefficient, the thermal conductivity and microhardness are listed for TiN, ZrN, $HfN_{0.86}$, $VN_{0.74}$, $VN_{0.93}$, $NbN_{0.5}$, NbN, $TaN_{0.5}$, TaN, $CrN_{0.5}$, CrN and $MoN_{0.5}$. The nitrides, in contrast to the metal which are hole conductors, show primarily electron conduction, as can be seen from the sign of the Hall coefficients of the nitrides except for $MoN_{0.5}$ are negative, and increase linearly with increasing acceptor properties of the incomplete 5d electron shell. There is a tendency to an increase in the negative thermoelectric coefficient with a decrease in nitrogen content when the higher nitride is a metallic conductor, and increases when the higher nitride is a semi-conductor as in the chrome-nitrogen system. The hardness of the mononitride is less than the hardness of the Me_2N compounds.

146. Samsonov, G. V., Koval'chenko, M. S. and Verkhoglyadova, T. S.
Preparation of the disilicides of refractory metals. RUSSIAN JOURNAL OF INORGANIC CHEMISTRY 4(12):1276–1279. 1959

The conditions for the preparation of the disilicides of ...Nb...Mo...by direct combination of the metals with silicon in an argon atmosphere were investigated. The following optimal conditions are recommended for making disilicides of sufficiently precise composition:...$NbSi_2$, 1000° for 0.5 hr.; ...$MoSi_2$, 1000° for 0.5 hr.;... The energies of activation of diffusion formation of the disilicide phases from metal powders and silicon were computed. The activation energy is higher in this case than in the formation of the same phase by reaction with compact metals, which is connected with the crystallization pressure on formation of intermetallides in powder mixtures. A supplementary investigation was made of the conditions for obtaining silicides of ...Nb...by thermal reduction of the oxides of the metals, using silicon, in a vacuum.

147. Schofield, T. H.
Melting point of niobium. INSTITUTE OF METALS. JOURNAL 85:372–374, Apr 1957.

Experimental redetermination finds melting point of columbium samples containing 0.12% gaseous impurities and 1.9% tantalum is 2468 ±10°C.

148. Searcy, A. W. and Tharp, A. G.
Dissociation pressures and the heats of formation of the molybdenum silicides.
JOURNAL OF PHYSICAL CHEMISTRY
64(10):1539–1542, Oct 1960.

Silicon partial pressures for dissociation of Mo_3Si, Mo_5Si_3 and $MoSi_2$ were measured by the Knudsen effusion method. Heats of dissociation at 298°K were calculated as 131.9, 131.1 and 117.2 kcal, respectively, and heats of formation as -23.5, -22.6 and -13.0 kcal. Data are tabulated.

149. Searcy, A. W.
Transition metals silicides and germanides.
In PROCEEDINGS. WADC CERAMIC CONFERENCE ON CERMETS. US. Air Force, Wright-Patterson Air Development Center, Ohio. WADC Technical Report No. 52-327. 1952. p. 275–279. ASTIA AD-1183.

150. Semmel, J. W., Jr.
The oxidation of tungsten and molybdenum from 1800 to 2500 F. In HIGH TEMPERATURE MATERIALS; CONFERENCE HELD IN CLEVELAND, OHIO, APRIL 16–17, 1957. Hehemann, R. F. and Ault, G. M., eds. N. Y., Wiley, 1959. p. 510–519

The oxidation of W between 1800 and 2500°F was studied and compared with Mo. Mo oxidized linearly between 1800 and 2500°F. The rate is insensitive to rate of air flow over a wide range, but Mo oxidizes rapidly in a direct air blast. The oxidation of W goes from linear to parabolic because of oxide melting at about 2200°F. Between 1800 and 2500°F, W oxidizes more slowly than Mo.

151. Shaffer, P. T. B.
DEVELOPMENT OF ULTRA REFRACTORY MATERIALS. Carborundum Co. Research and Development Div., Niagara Falls, N.Y. Summary report, 1 Nov 1959 through 31 Oct 1960. 15 Dec 1960. 39p. [Contract NOrd-17175].

ZrB -10$MoSi_2$ was tested to determine its physical and chemical properties. The composition has good oxidation resistance. Prd iminary studies were made in mixed carbide systems of the Group IVa and Va metals.

152. Shaffer, P. T. B.
DEVELOPMENT OF ULTRA REFRACTORY MATERIALS. Carborundum Co., Research and Development Div., Niagra Falls, N.Y. Progress Report No. 24. 28 Apr 1961. 12p. [Contract NOrd-17175]

Melting points of molybdenum sheet and ct ZrC and 4TaC·ZrC bars were measured using the apparatus developed.

153. Shaffer, P.T.B., Sr.
An oxidation resistant boride composition. AMERICAN CERAMIC SOCIETY. BULLETIN 39(9):469, Sep 1960. (abstract)

A modified ZrB_2 composition with good resistance to oxidation and thermal shock has been developed. A densified mixture of ZrB_2-5-15% $MoSi_2$ has good thermal and electrical conductivity and good high temperature strength. The composition may be used as a mass or as a coating on other ceramics to confer oxidation resistance to 1950°C. This paper was presented before the Fall Meeting of the Refractories Division, American Ceramic Society in Bedford Springs, Pa., 6–8 October 1960.

154. Sibert, M. E. and Steinberg, M. A.
Preliminary study of the equilibrium of carbon and oxygen in columbium with carbon monoxide above 1600°C. In REACTIVE METALS; PROCEEDINGS OF THE THIRD ANNUAL CONFERENCE SPONSORED BY NIAGARA FRONTIER SECTION IN COOPERATION WITH THE METALLURGICAL SOCIETY, AMERICAN INSTITUTE OF MINING, METALLURGICAL, AND PETROLEUM ENGINEERS, BUFFALO, NEW YORK, MAY 27–29, 1958.
Clough, W. R., ed. New York, Interscience, 1959.
p. 171–179. (Metallurgical Society Conferences, v. 2).

A preliminary study of the equilibrium between CO and dissolved C and O in columbium has been made.

$$C(Cb) + O(Cb) \rightleftharpoons CO/Cb$$

The study was carried out by use of a high-temperature graphite resistor heated furnace. Samples of impure Cb were lowered into the evacuated hot zone and allowed to reach equilibrium, the furnace unit being isolated from the pumping system. Assuming C and O in Cb to follow the behavior of ideal solutions, approximate equilibrium constants have been derived based on the equilibrium pressure and analysis of Cb samples after equilibrium. ΔF values for the reaction have been obtained. The work covers the temperature range 1600–1900°C. A linear relationship of log K^1 vs. $1/T$ indicates the basic assumption to be valid. Equipment, procedures, and results are fully described.

155. Sibert, M. E., Kolk, A. J., Jr. and Steinberg, M. A.
A review of the extractive metallurgy of niobium.
In TECHNOLOGY OF COLUMBIUM (NIOBIUM)
N.Y., Wiley, 1958. p. 20–34.

Chemical reduction of oxides and halides such as Cb_2O_5, K_2CbOF_5, K_2CBF_7 and $CbCl_5$ has been conducted with varying degrees of success. Aqueous electrochemical reduction has not yet yielded a metallic deposit. The electrolysis of molten baths containing K_2CbOF_5 or K_2CbF_7 has produced Cb metal. The more promising methods of preparation of Cb metal at the present include reaction of Cb_2O_5 with C, the H_2 and active-metal recution of $CbCl_5$, and electrolysis of K_2CbF_7-NaCl melts.

156. Sikora, P. F.
HIGH-TEMPERATURE TENSILE AND STRESS-RUPTURE PROPERTIES OF SOME ALLOYS IN THE TUNGSTEN-MOLYBDENUM SYSTEM.
National Aeronautics and Space Administration, Washington, D. C. Technical note D-1087.
Apr 1962. 19p. ASTIA AD-274 567.

Three alloys consisting of 10, 25, and 50 st-% Mo in W were evaluated at 2500 to 4400 F to determine their tensile properties. Results showed the 10 and 25wt-% alloys have higher tensile strength than unalloyed W at 2500 and 3000°F and to unalloyed W from 2500 to 3200°F. Unalloyed W has higher strength than the alloys above 3500°F. The results of stress-rupture tests for the 50 wt-% alloy are presented for the temperature range of 2500 to 3500°F in the as-worked and fully annealed conditions.

157. Simnad, M. and Spilners, A.
Kinetics and mechanism of the oxidation of molybdenum. JOURNAL OF METALS 7:1011–1016, Sep 1955.

Rates of formation of different oxides on molybdenum, in pure oxygen at one atmosphere pressure, have been determined between 500 and 770°C. They grow almost entirely by diffusion of oxygen anions.

158. Sims, C. T., Klopp, W. D. and Jaffee, R. I.
Studies of the oxidation and contamination resistance of binary columbium alloys.
AMERICAN SOCIETY FOR METALS. TRANSACTIONS 51:256–281, 1959.

A study of the effects of binary alloying additions on the oxidation and contamination resistance of columbium has been conducted. The alloys contained up to 35 a/o titanium, chromium, and zirconium, 25 a/o vanadium, molybdenum, tantalum and tungsten, and 5 a/o beryllium, boron, cobalt, iron, manganese, nickel, and silicon. The oxidation and contamination studies were conducted in air at 600, 800, and 1000°C (1110, 1470 and 1830°F.) Four elements, titanium, vanadium, molybdenum, and chromium improved oxidation resistance. The concentrations for optimum oxidation resistance at 1000°C (1830°F) were 25 a/o titanium, 10 a/o vanadium, 5 a/o molybdenum, and 15 a/o chromium. Titanium was most effective in reducing the rate of

oxidation; at 600 to 1000°C (1110 to 1830°F), the columbium-25 a/o titanium alloy oxidized one-tenth to one-twentieth as rapidly as pure columbium. Contamination was investigated by hardness penetration measurements on all oxidized alloys except those containing beryllium or boron. Zirconium and titanium markedly reduced the depth of oxygen contamination resulting from exposure to air. Diffusion coefficients for oxygen in the columbium binary alloys were calculated, and related by activation-energy plots where possible. Zirconium, titanium, chromium, and vanadium were most effective in reducing oxygen diffusion into columbium.

159. Smith, W. H. and Grenoble, H. E.
TREATMENT OF STEAM FOR THE PROTECTION OF CERTAIN METAL PARTS. (Assigned to General Electric Co.) U.S. Patent No. 3,032,386. 1 May 1962.

Molybdenum or its alloys may be used in a 1000°F atmosphere containing steam if the atmosphere also contains sufficient hydrogen to prevent trioxide formation but not dioxide formation.

160. Speiser, R., Blackburn, P. and Johnston, H. L.
Vapor pressure of niobium. ELECTROCHEMCIAL SOCIETY. JOURNAL 106:52–53, Jan 1959.

Langmuir method used with an accommodation coefficient of unity assumed. Measurementsof rate at which a Cb surface sublimed into a high vacuum. Equation for the heat of sublimation at absolute zero.

161. Staskiewicz, B. A., Tucker, J. R. and Snyder, P. E.
The heat of formation of molydenum dioxide and molybdenum trioxide. AMERICAN CHEMICAL SOCIETY. JOURNAL 77(11):2987–2989, 5 Jun 1955.

The heats of combustion of molybdenum and MoO_2 were studied; with the metal combustion yielded only a mixture of di- and trioxides. Three heat values were derived from the data: the standard heat of formation of MoO_3 is -744.7 int. kjoules; the standard heat of formation of MoO_2 to MoO_3 is -155.3 int. kjoules. Data are tabulated.

162. Steinitz, R.
Materials based on intermetallic compounds.
In HIGH TEMPERATURE MATERIALS.
Hehemann, R. F. and Ault, G. M., eds.
N.Y., Wiley, 1959. p. 182–189.

Systems between transition metals were studied to investigate the properties of their intermetallic compounds. The requirements of a congruent melting point above 15[illegible]0°C and of a fair oxidation resistance eliminated most... Mo_3Si_2-Mo_3Si-$MoSi_2$ was promising.

163. Syre, R.
NIOBIUM, MOLYBDENUM, TANTALUM AND TUNGSTEN. A SUMMARY OF THEIR PROPERTIES WITH RECOMMENDATIONS FOR RESEARCH AND DEVELOPMENT. Advisory Group for Aeronautical Research and Development, Paris. 1961. 130p. (AGARDograph 50.) ASTIA AD-274 036

The metals Nb, Mo, Ta and W are discussed from the aspects of: (1) physical properties, (2) mechanical characteristics at various temperatures (unalloyed metal and main alloys), and (3) oxidation behavior. Reference is also made to the problems of protective coatings, joining and fabrication. Following the examination of the present state of knowledge, recommendations are made for research and development. A detailed study is presented of the state of knowledge of each of the metals in regard to phase diagrams, impurities (determination and effects), diffusion couples, behavior at high temperature under exposure to different gases, and protective coatings. A bibliography of the studies of the 4 metals and their alloys is also presented.

164. Tamura, K.
Physical and mechanical properties of $MoSi_2$ base heating elements. FUNTAI OYOBI FUNMATSUYAKIN 8(3):113–120, 1961 (In Japanese)

The results of a corrosion test of an $MoSi_2$ electric heating element in various gases and of mechanical tests at room and high temperature are summarized as follows: (1) Oxidation in air amounted to 0.0001 g/cm^2 at 1200°C for 1000 hr, 0.0015 g/cm^2 at 1300°C for 360 hr, and 0014 g/cm^2 at 1400°C for 96 hr. (2) Amounts of corrosion in various gases at 1200°C were 0.0004 g/cm^2/day in H_2 0.0003 g/cm^2/day in N_2,

0.0005 $g/cm^2/day$ in NH_3, 0.0010 $g/cm^2/day$ in CO_2, and 0.0001 $g/cm^2/day$ in vacuum at 1600°C. (3) Tensile strength, compressive strength, and modulus of rupture at room temperature were 15.4 kg/mm^2, 12.5 kg/mm^2, and 16.1 kg/mm^2, respectively. (4) Modulus of rupture at high temperature decreased as the atmospheric temperature increased, e.g., when the latter increased from room temperature to 1250°C., the former decreased from 16.1 kg/mm^2 to 9.5. (5) Tensile strength at 1600°C was 1 kg/mm^2. (6) Values of bending creep at 1200°C were 1.40 mm at 4 kg/mm^2 load and 0.42 mm at 1 kg/mm^2 load.

165. Tamura, K. and Kawaguchi, T.
Sintering of $MoSi_2$ heating element and its application. FUNTAI OYOBI FUNMATSUYAKIN 7(4):156–162, 1960. (In Japanese)

Preliminary experiments were made to prepare an electric heating element composed chiefly of $MoSi_2$ fine powder mixed with oxide powders (Al_2O_3, SiO_2), and the mixture was hot-pressed and sintered. The optimum sintering conditions were thus obtained. The specimens were subjected to physical and mechanical tests, and the results are summarized as follows: (1) The sintering temperature of $MoSi_2$ with oxides (Al_2O_3), SiO_2) is 1600° to 1650°C, and the sintering pressure is 250 kg/cm^2, the density of the specimen being 90 to 97% of the theoretical density. (2) Corrosion resistance increases with increasing amount of oxides added. (3) The vickers hardness of $MoSi_2$ with oxide admixture is higher than that of pure $MoSi_2$, ranging from 971 to 1300. In general, Vickers hardness at high temperatures decreases as the atmospheric temperature increases. (4) The addition of oxides to $MoSi_2$ raises the electrical resistance of the specimen. (5) Electrical resistance increases linearly with rise in temperature, e.g., 0.1×10^{-2} Ω-cm at 1500°C. (6) Modulus of rupture decreases with the increasing amount of the oxides. (7) Thermal shock resistance increases with the addition of Al_2O_3. (8) The oxidizing tests in the air at 1200°C proved successful. The increase in weight of the specimen is in the range 0.0002 to 0.0005 $g/cm^2/day$.

166. Taylor, R. E. and Finch, R. A.
THE SPECIFIC HEATS AND RESISTIVITIES OF MOLYBDENUM, TANTALUM, AND RHENIUM FROM LOW TO VERY HIGH TEMPERATURES. Atomics International, Div. of North American Aviation, Inc., Canoga Park, Calif. Report NAA-SR-6034. 15 Sep 1961. 37p. [Contract AT(11-1)-GEN-8]

The specific heat of molybdenum was measured in the 200°–2880°K range using the advanced equipment shown. Results agree well with previous work. Specific heat does not saturate at the Dulong-Petit value and the electronic contribution is considerable. Vacancy formation does not account for the upswing near the melting point. The electrical resistivity in molybdenum does not change much on heating to high temperatures. Graphs are shown. 21 references.

167. Tietz, T. E. and Wilson, J. W.
MECHANICAL OXIDATION AND THERMAL PROPERTY DATA FOR SEVEN REFRACTORY METALS AND THEIR ALLOYS. Lockheed Missiles and Space Div., Sunnyvale, Calif. Final Report. LMSC Report No. 2-36-61-1. 15 Sep 1961. 315p. [Contract NOas-60-6119-c]

Property data for Cr, Nb, Mo, Re, Ta, W., and V are presented in detail. The figures presented are intended to serve only as a general guide, as the properties are sensitive to variations in impurities, fabrication history, thermal treatments, and specific test conditions. Details regarding the known effects of these variables are discussed. The melting point, crystal structure, and density of the seven refractory metals are summarized in tabular form. The mechanical, oxidation and thermal properties of these metals and/or their alloys are summarized from comparative data presented for each metal.

168. Tottle, C. R.

Niobium and its alloys. NUCLEAR ENGINEERING

3:212–216, May 1958.

Niobium has much to offer in the field of liquid-cooled reactors not involving oxidizing conditions, particularly where high temperatures are involved. The extraction process is tedious, but manufacture is comparatively free from severe troubles arising from peculiar properties. The production and fabrication of the metal and its alloys will always be expensive owing to the necessity for protection from atmospheric attack during sintering, melting, heat treatment, and joining. Future development should simplify techniques and improve the properties in such a way that the cost is justified by the behavior in service. Tables of all the available properties of niobium are presented.

169. Tottle, C. R.

The physical and mechanical properties of niobium.

INSTITUTE OF METALS. JOURNAL

85:375–378, Apr 1957.

The possible employment of Nb or its alloys in reactor components is partially dependent on its possessing suitable physical and mechanical properties. It was therefore necessary to assemble accurate data, covering the range of temperatures involved in reactor operation, on thermal expansion, thermal conductivity, and tensile and creep properties, using metal of a suitable standard of purity for fabrication into complex shapes. Experimental methods already established for metals readily attacked by atmospheric gases were employed. Thermal conductivity is shown to increase, and tensile properties to decrease relatively slowly, with rise in temperature. Tensile properties are markedly affected by small additions of oxygen and creep measurements are susceptible to serious error if oxygen gains access to the system during long periods of test. Reference is made to preliminary results on the effect of alloying elements.

170. Truesdale, R. S. et al
INVESTIGATION OF THE EFFECTS OF PROCESSING VARIABLES AND FABRICATION TECHNIQUES UPON THE PROPERTIES OF INTERMETALLIC COMPOUNDS. Directorate of Materials and Processes, U.S. Air Force Systems Command, Aero. Systems Div., Wright-Patterson Air Base, Ohio.
Report ASD TDR 62-476. Jun 1962. 267p.

Experimental investigation of the sintering of Nb_2Be_{17} fabricated by uniaxial pressing and sintering techniques. The effects of particle size, stoichiometry, temperature, and time upon the sintered density, grain size, and strength of the compound are described. A modulus-of-rupture evaluation is made on Nb_2Be_{17} up to 2,750°F., and results of tensile, Young's modulus, thermal conductivity, and oxidation studies are discusses. The metallurgical fabrication techniques of forging, forming and rolling as applied to the compound are described.

171. Tye, R. P.
Preliminary measurements on the thermal and electrical conductivities of molybdenum, niobium, tantalum and tungsten. JOURNAL OF THE LESS-COMMON METALS 3(1):13–18, Feb 1961.

The thermal conductivity of molybdenum in the 50°–350°C range and electrical resistivity in the 20°–1450°C range were measured. Heat treatment does not affect thermal properties. Relation to results of previous workers and to the Lorenz function are discussed. Data are tabulated.

172. United Kingdom Atomic Energy Authority. Industrial Group. Dounreay, Caithness, Scotland.
ON THE COEFFICIENTS OF LINEAR THERMAL EXPANSION OF PURE NIOBIUM BETWEEN 60°K and 1200°K. Report IGR-TM/D-073.
Feb 1958. 5p.

173. U.S. National Bureau of Standards, Washington, D. C. THERMAL CONDUCTIVITY AND HEAT CAPACITY PROJECT. Progress Report no. 11 for 7 Nov 1953 to 6 Feb 1954. Report NBS-3179. Mar 1954. 8p.

Data on the thermal conductivity of $MoSi_2$ are presented graphically and in tabular form.

174. Vogel, R. and Gerhardt, R. The iron-molybdenum-silicon system. ARCHIV FUER DAS EISENHUETTENWESEN 32(1):47-56 Jan 1961. (In German)

Thermal and metallographic investigations made it possible to construct for the first time the phase diagram of the Fe-Mo-Si system. An equilibrium line was found between FeSi and $MoSi_2$. Nine crystal phases were found in the system:... Fe_2MoSi_2, Mo_3Si, Mo_3Si_2 and $MoSi_2$. Reactions observed include six transition equilibria (of the type melt + A (solid) $\rightleftharpoons$ B(solid) + C(solid), two euctectic and one eutectoid reaction. The investigations revealed also three ternary phases: Fe_2MoSi_2, Fe_3Mo_2Si and Fe_2Mo_2Si. Phase diagrams and micrographs are shown.

175. Walker, B. E., Grand, J. A. and Miller, R. R. High-temperature heat content and heat capacity of Al_2O_3 and $MoSi_2$. JOURNAL OF PHYSICAL CHEMISTRY 60:231-233. 1956.

The heat capacity of Al_2O_3 and $MoSi_2$ were derived from heat content measurements made over the range 30 to 700° for Al_2O_3, and 30 to 900° for $MoSi_2$. The changes in enthalpy were determined by the "drop" method and a copper-block calorimeter. The calorimeter is a modified version of one previously used and described by J. C. Southard. The furnace for heating the samples is specially constructed for high temperature heat capacity work.

176. West, E. D., Ditmars, D. A. and Ginnings, D. C. THERMAL AND RELATED PHYSICAL PROPERTIES OF MOLTEN MATERIALS. PART 5. THE THERMAL CONDUCTIVITY AND HEAT CAPACITY OF MOLYBDENUM DISILICIDE. U.S. National Bureau of Standards, Washington, D. C. WADC Technical Report No. 53-201, Pt. 5. 1954. 19p. [Contract AF33(616)52-10] ASTIA AD-49 098.

A new apparatus is described for determining at high temperatures the thermal conductivities of liquids and solids having relatively high conductivities. The apparatus employs steady state longitudinal heat flow along a rod surrounded by a matched guard tube. Results on molybdenum disilicide are described.

177. Westinghouse Alloy B 33 (high temperature columbium alloy). ALLOY DIGEST Cb-1, p. 1–2, Aug 1961.

Physical and mechanical properties, workability, machinability and weldability information and applications are given for a high temperature corrosion resistant Cb alloy containing 5.0% vanadium.

178. What can resist re-entry heat? MISSILES AND ROCKETS. 5(32):24–25, 3 Aug 1959.

$MoSi_2$-coated Mo-0.5Ti-0.07Zr is a promising material for the leading edge of wings of hypersonic vehicles. Advantages are high thermal conductivity, low thermal expansion, recrystallization temperature of 2900°F, good short-time ultimate tensile strengths at 2400° and 3000°F, and, with the coating, high oxidation resistance. Comparisons are made with a number of other materials tested.

179. Wilcox, B. A.
Effect of hydrogen on dislocation locking in niobium. JOURNAL OF THE LESS COMMON METALS 2:292–303, Apr–Aug 1960.

Strain-aging tendencies of as-received commercial purity arc-melted Cb and hydrogenated Cb from the same heat were investigated using yield point return and dynamic modulus measurements to study the aging process. Comparison of activation energies for strain aging with those for interstitial diffusion revealed that H could be responsible for dilocation locking in Cb.

180. Wood, W. D., Deem, H. W. and Lucks, C. F.
THE EMITTANCE OF CHROMIUM, COLUMBIUM, MOLYBDENUM, TANTALUM, AND TUNGSTEN. Defense Metals Information Center, Battelle Memorial Institute, Columbus, Ohio. DMIC-Memo-141. 10 Dec 1961. 48p.

A compilation is presented of the emittance, reflectance, and absorbance of Cr, Nb, Ta, W, Nb-10% Mo-10%Ti, and Mo-0.5% Ti, as functions of temperature and wavelength. Both total and spectral values for these properties are given, and the effects of surface conditions are taken into account. A bibliography of 24 references is included.

181. Worthing, A. G.
Physical properties of well seasoned molybdenum and tantalum as a function of temperature. PHYSICAL REVIEW 28:190–201, Jul 1926.

Spectral emissivity, spectral reflectivity, radiation intensity, and thermal expansion measurements as a function of temperature have been made for well seasoned molybdenum and tantalum. From the data thus obtained values for many other physical quantities as functions of temperature have been computed. As such physical quantities, average visible emissivity, color emissivity, total emissivity, brightness temperature, solar temperate, radiation temperature, normal brightness, luminous efficiency, and certain temperature variation coefficients have been included. Comparison measurements on moderately pure and very pure molybdenum showed no appreciable variation

in radiation properties, though appreciable variations appeared in their resistivities. The results for very pure molybdenum and moderately pure tantalum are incorporated in tables giving values where known for every 200 degrees between room temperature and melting points.

182. Worthing, A. G.
The temperature scale and the melting point of molybdenum. PHYSICAL REVIEW 25:846–857 Jun 1925.

Spectral emissivities and temperature scales of Mo from 300° to 2900°K. A tubular filament of pure Mo was made by winding ribbon in a close helix about 15 mm long and 1 mm in diam. Spectral emissivities were then obtained by observing the ratio of the outer surface brightness to the interior brightness as seen through small holes in the wall. The results were checked by two other methods. At room temperature the spectral reflectivities were determined and found to be independent of previous heat treatment. For 0.665 μ the emissivities at 300°, 1500° and 2800°K are 0.419, 0.371 and 0.331; for .475 μ, 0.424, 0.390 and 0.365 each within ± .007. These values are all lower than those reported by others, probably because of the greater purity of the Mo or of the better black body conditions. Hence these are believed more reliable. These emissivities lead to a difference between true temperature and brightness temperature as a function of the brightness temperature of 46° at 1000°, 113° at 1500°, 217° at 2000°, and 370° at 2500°K. Melting point of Mo has been obtained by observing the brightness temperature of a Mo V-filament at melt and applying the correction to true temperature, and by two other methods. The rounded average of 97 reading yields 2895°K±10 (c_2 = 14,330 μ deg., T_{Au} = 1336°K). This value is higher than most of the results of others.

183. Wright, R. W.
Positive and negative thermionic emission from molybdenum. PHYSICAL REVIEW 60:465–467, 15 Sep 1941.

The positive and negative thermionic emission from molybdenum have been studied. The electron work function was found to be 4.20 volts and the positive ion work function was 8.6 volts. The positive ion constant term was in good agreement with Smith's equation.

184. Zmeskal, O. and Brey, M. L.
Oxidation of zirconium-columbium alloys in oxygen at 525–1090°C. AMERICAN SOCIETY FOR METALS. TRANSACTIONS 53:415–431, 1961.

Alloys of high-purity zirconium and columbium were heated in oxygen at pressures lower than atmospheric and temperatures ranging from 525 to 1090°C. Rates of oxidation at constant pressure and temperature were measured by a volumetric procedure for periods of 2 to 6 hrs. X-ray diffraction patterns were made of the free oxides. At 600°C the oxidations conformed approximately to the parabolic rate law during the early stages of oxidation; at 900° the general relationship, w^n-kt, was not followed by any of the alloys in the oxidation process, but all appeared to increase gradually in rate toward a linear relationship. At 1090°C values of n ranging from about 3 for alloys low in columbium to 2 for alloys high in columbium remained approximately constant during the period of measurement.

AUTHOR INDEX